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Synthesis and biological evaluation of gramicidin S dimers

Grotenbreg, Gijsbert M.; Witte, Martin D.; Hooft, Peter A.V. van; Spalburg, Emile; Reiß, Philipp; Noort, Daan; Neeling, Albert J. de; Koert, Ulrich; Marel, Gijsbert A. van der; Overkleeft, Herman S.

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Supporting Information for

Synthesis and biological evaluation of gramicidin S dimers

Gijsbert M. Grotenbreg, Martin D. Witte, Peter A. V. van Hooft, Emile Spalburg,
Philipp Reiß, Daan Noort, Albert J. de Neeling, Ulrich Koert, Gijsbert A. van der Marel,
Herman S. Overkleeft and Mark Overhand

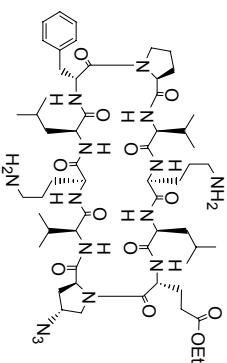
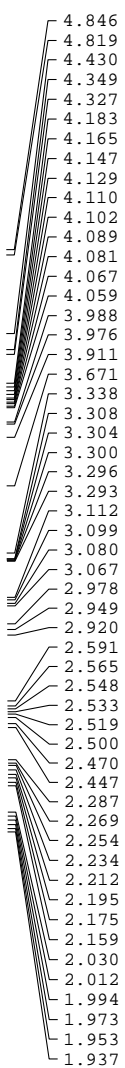
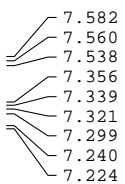
Selected NMR spectra [‡]

(in CD₃OH, at 298 K, at 400 MHz)

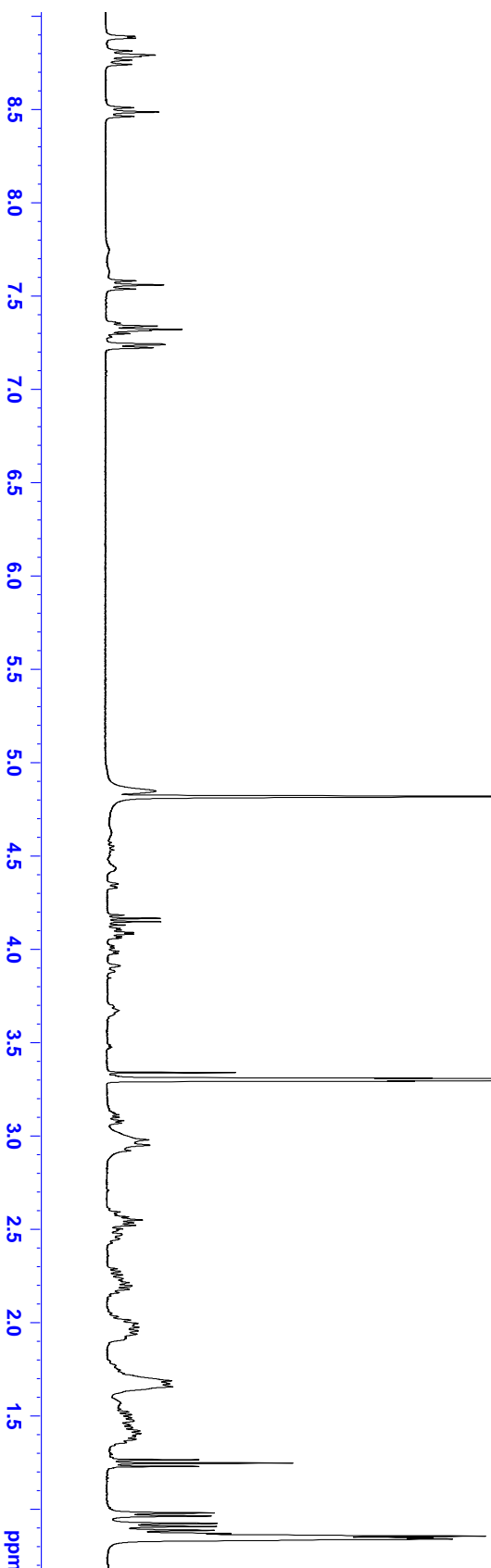
Compound 13	¹ H	Page 2-5
Compound 13	TOCSY	Page 6,7
Compound 14	¹ H	Page 8-11
Compound 14	TOCSY	Page 12,13
Compound 17	¹ H	Page 14-17
Compound 18	¹ H	Page 18-21
Compound 21	¹ H	Page 22-25
Compound 22	¹ H	Page 26-29
Compound 23	¹ H	Page 30-33
Compound 23	TOCSY	Page 34-36

[‡] Variation in the experimental conditions, such as the solvent system or temperature, could not resolve the severe overlap of resonances in the ¹H NMR spectra due to the high degree of symmetry present in the gramicidin S analogues and the dimers thereof.

mwe1101 (epje 1) 298 K

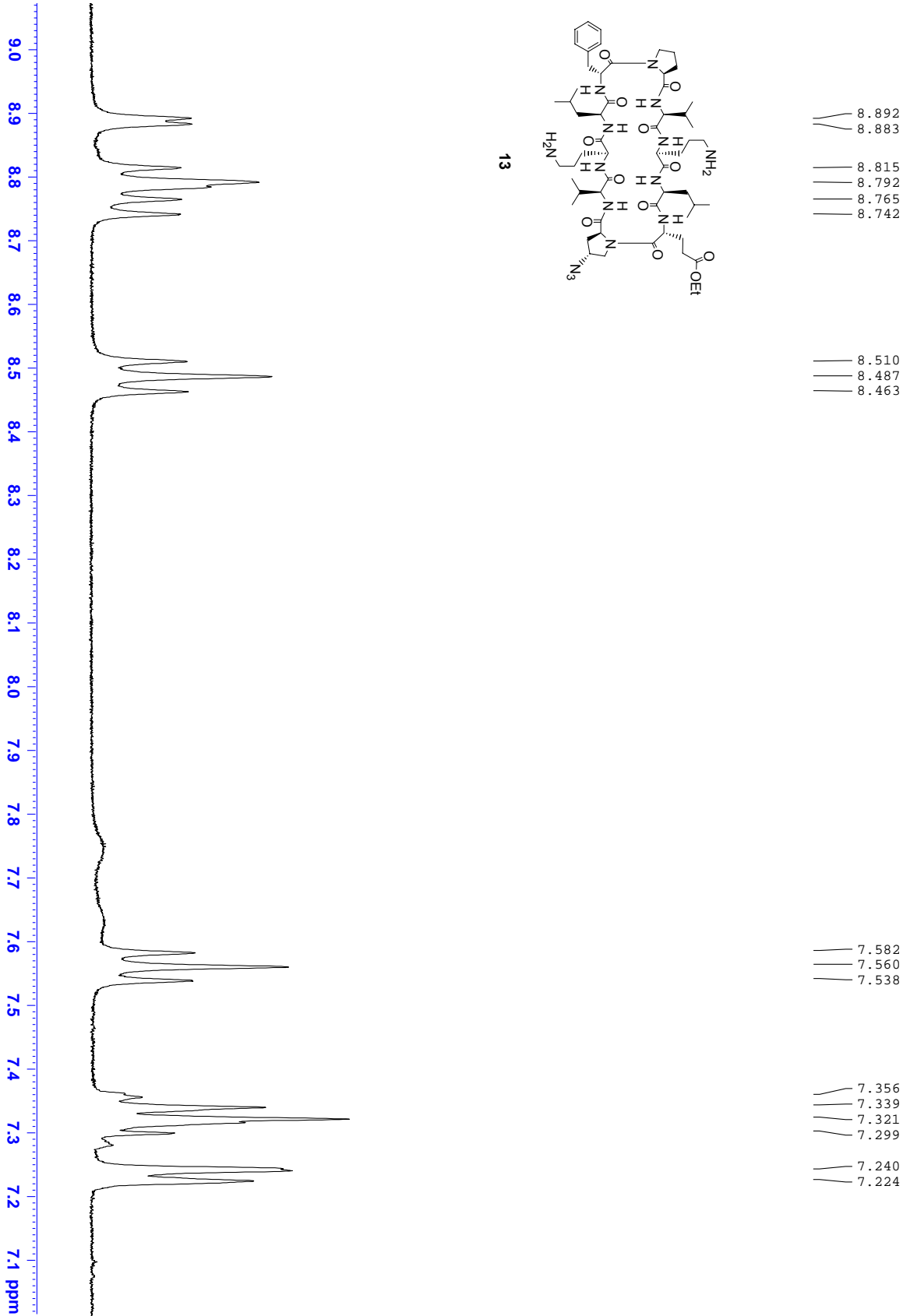


13



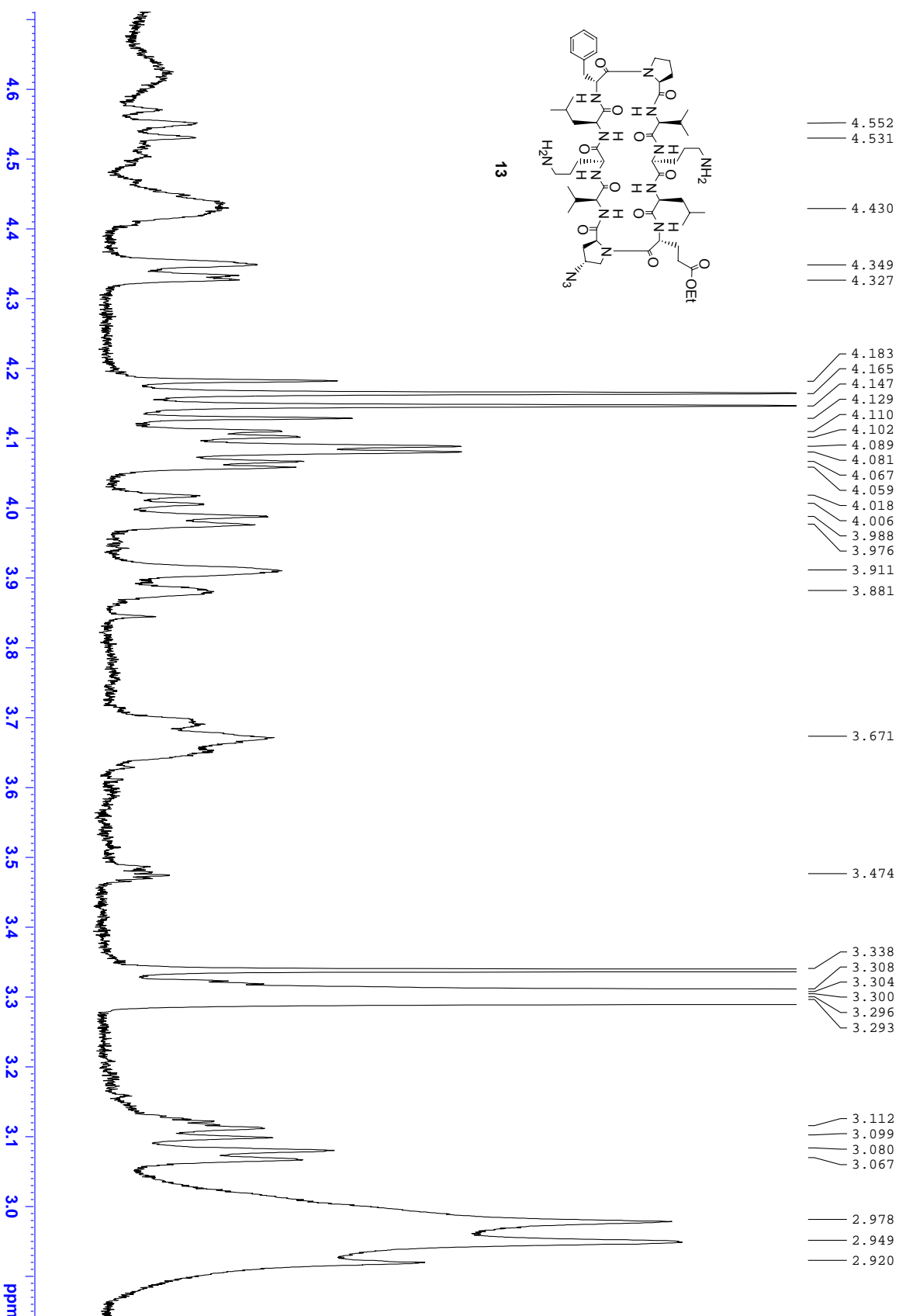
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TD	1228		
SOLVENT	Me2D		
DS	64		
NS	0		
SWH	3720.238 Hz		
FIDRES	0.193480 Hz		
AQ	2.5842931 sec		
RG	161.3		
DW	134.400 usec		
DE	38.50 usec		
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DI2	0.00002000 sec		
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NUC1	1H		
P1	7.30 usec		
P11	4.00 dB		
P19	52.29 dB		
SFO1	400.1319450 MHz		
F2 - Processing parameters			
SF	400.1300114 MHz		
SF1	37378		
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SSB	0		
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PC	1.00		

mwe1101 (epje 1) 298 K



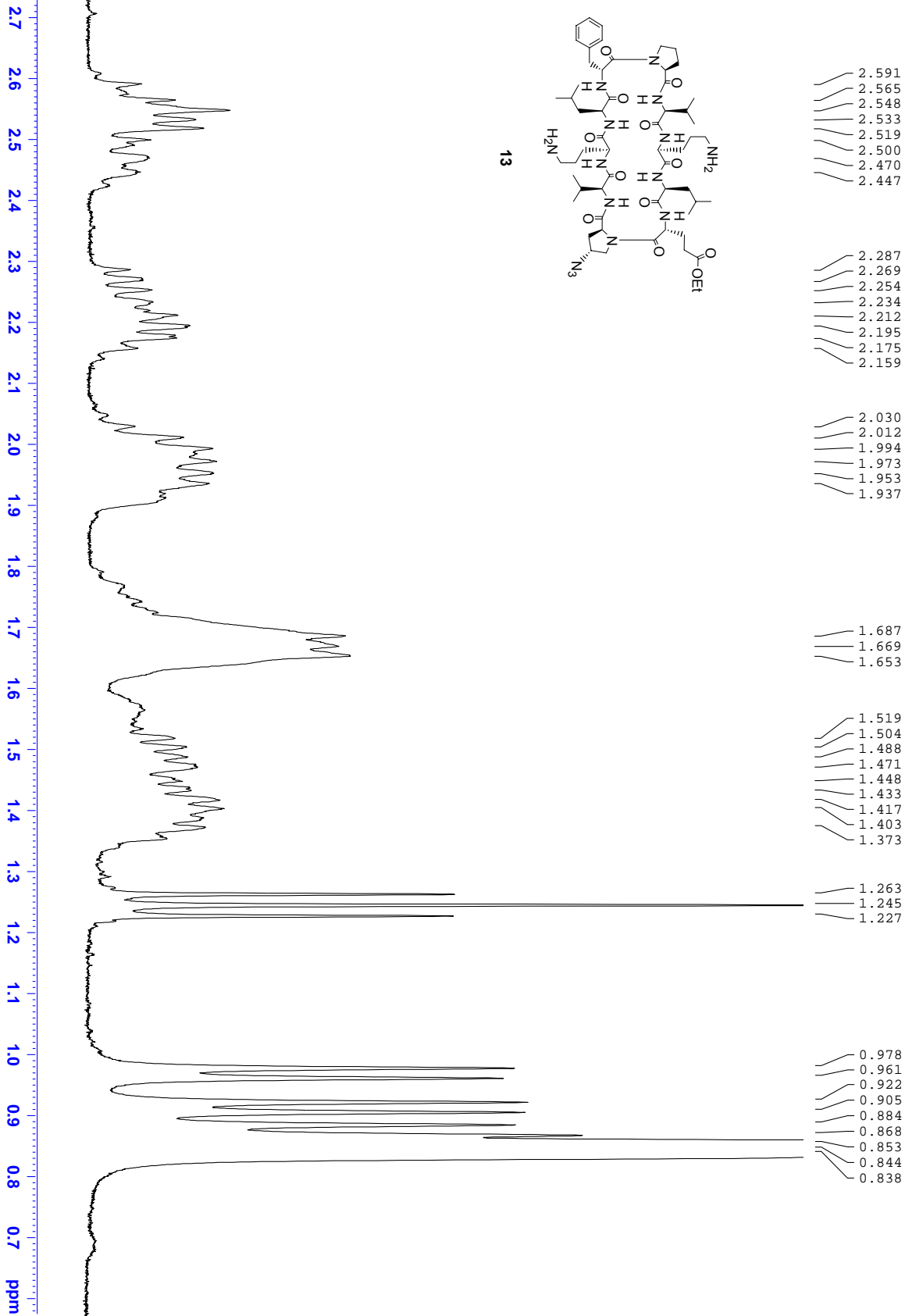
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RG 161.3
DW 134.400 use
DE 300.0 K
TE 1.0000000 sec
d12 0.0000000 sec
d13 0.0000000 sec
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P1 7.30 use
PL1 4.00 dB
PL9 52.29 dB
SFO1 400.1319405 MHz
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GB 0.4
PC 1.00

mwe1101 (epje 1) 298 K



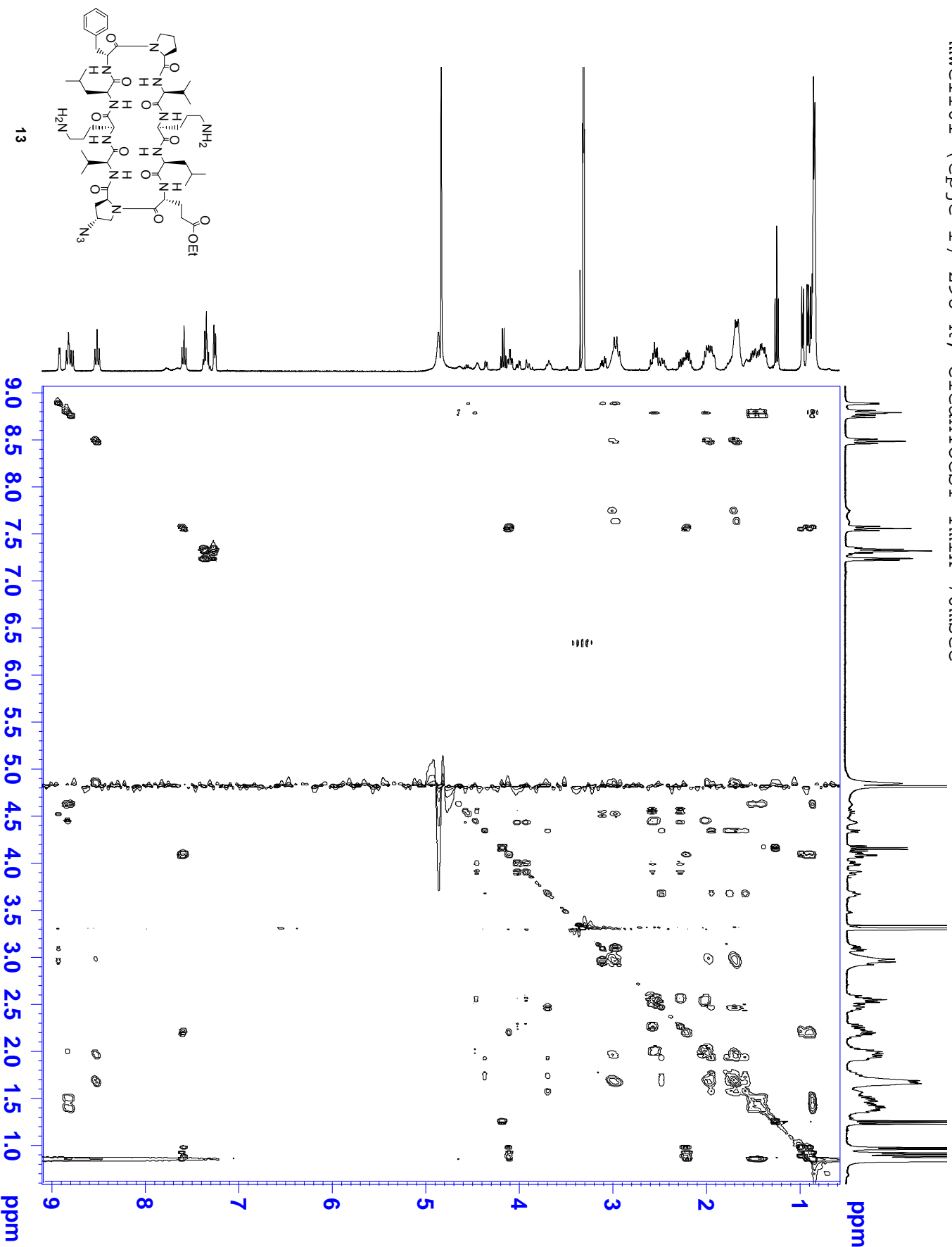
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TD 19228
SOLVENT MeOD
NS 64
DS 0
SWH 3720.238 Hz
FIDRES 0.193480 Hz
AQ 2.5842931 sec
RG 161.3
DW 134.400 usec
DE 8.50 usec
TE 300.0 K
D1 1.00000000 sec
d12 0.00000000 sec
d13 0.00000300 sec
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P1 7.30 usec
PL1 4.00 dB
PL9 52.29 dB
SFO1 400.1319405 MHz
F2 - Processing parameters
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SF 400.1300114 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.4
PC 1.00

mwel101 (epje 1) 298 K



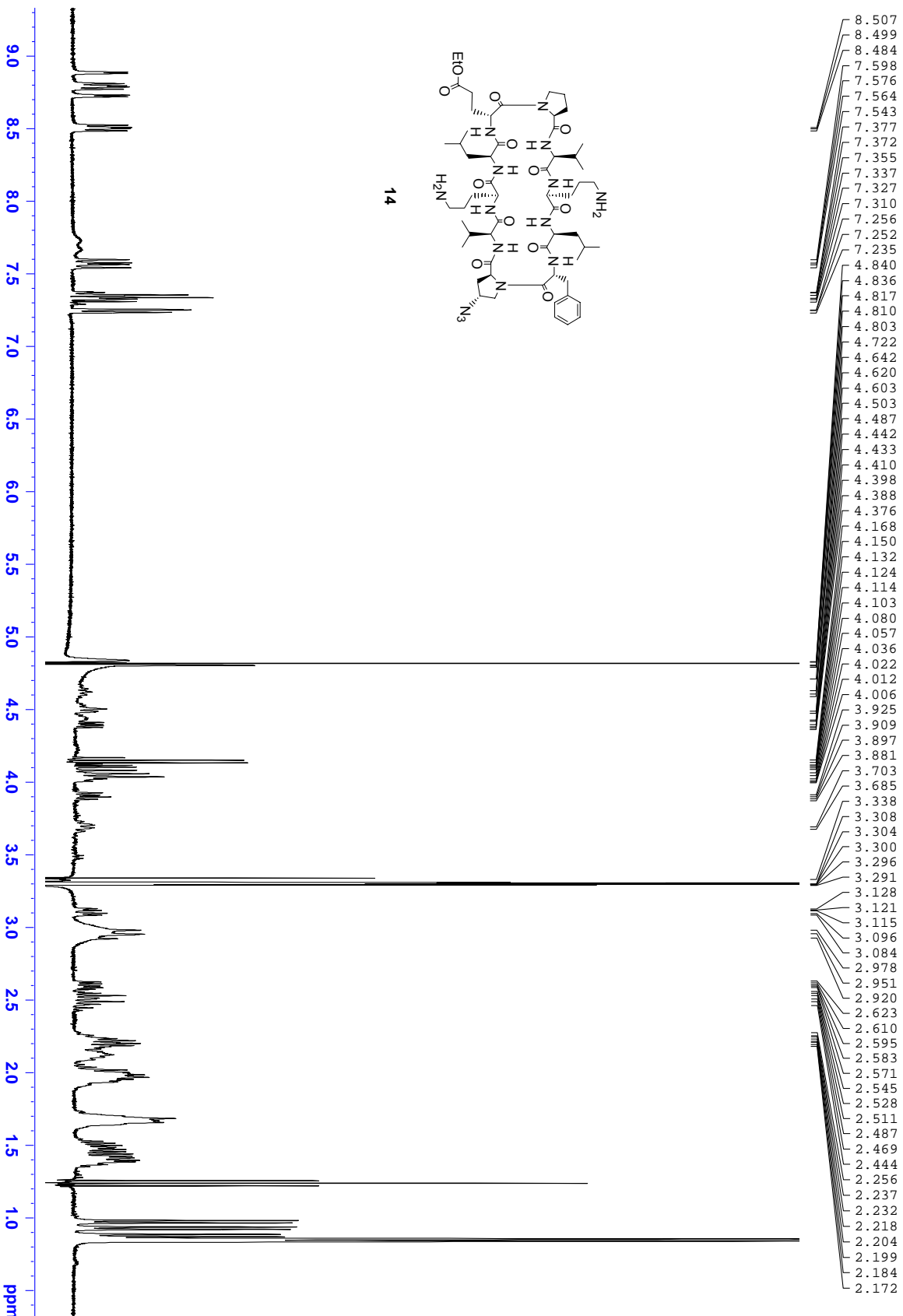
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PROCNO 1
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PULPROG zgpg
TD 13228
FIDRES 0.193480 Hz
RG 161.3
DW 134.400 us
TE 300.0 K
D1 1.00000000 sec
d12 0.00002000 sec
d13 0.00000300 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 us
PL1 4.00 dB
PL2 52.29 dB
SFO1 400.1319405 MHz
F2 - Processing parameters
SI 32768
SF 400.1300114 MHz
WDW GM
SSB 0
LB -0.20 Hz
GB 0.4
PC 1.00

mwel101 (epje 1) 298 K, cleanTOCSY Tmix=70msec



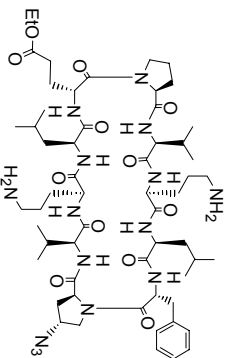
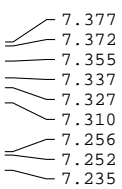
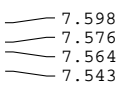
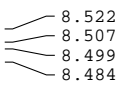
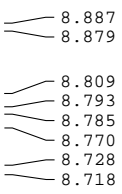
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NAME	gsjso42003
EXPNO	9
PROCNO	1
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Time	18.34
INSTRUM	5 mm
PROBHD	ABX1-HB-BB
PULPROG	zgpg30
TD	65536
NOFWDFT	1
DS	32
SWH	256
FIDRES	3720.238 Hz
AQ	1.816522 Hz
RG	0.2753012 sec
DW	2048
DM	134.400 usec
DE	8.50 usec
TE	300.0 K
d0	0.00000300 sec
d1	1.00000000 sec
d12	0.03000000 sec
d13	0.00000000 sec
d14	0.00001400 sec
d15	0.00006500 sec
D20	0.00026880 sec
IN0	18
MOREST	0.00000000 sec
MCWK	0.00939939 sec
SOLVENT	256
===== CHANNEL f1 =====	
NUC1	1H
F1	6.10 usec
P1	15.00 usec
P2	25.00 usec
P3	50.00 usec
P7	2500.00 usec
P17	4.00 dB
P19	52.29 dB
P10	16.50 dB
SFO1	400.1319388 MHz
F1 - Acquisition Parameters	
NU0	1
TD	512
SFO1	400.1319388 MHz
FIDRES	7.2.928 Hz
SW	9.298 ppm
PRMODE	States-TPII
F2 - Processing Parameters	
SI	2048
SF	400.1300114 MHz
WDW	OSINE
SSB	2.3
LB	0.00 Hz
GB	0
PC	1.00
F1 - Processing Parameters	
SI	1024
MC2	States-TPII
SF	400.1300064 MHz
WDW	OSINE
SSB	2.1
LB	0.00 Hz
GB	0

mwc1101 (epje 1) 298 K

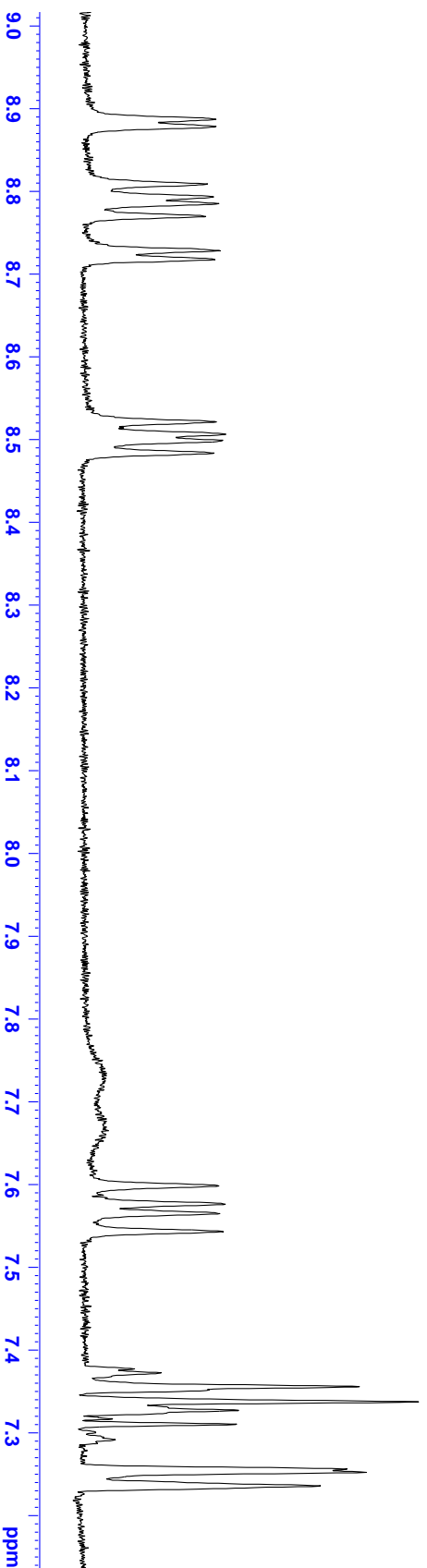


Current Data Parameters	
NAME	gljs042003
EXPNO	6
PROCNO	1
F2 - Acquisition Parameters	
Date_	20030405
Time	13.41
PROBHD	5 mm BBI 1H-2B
PULPROG	zgpg
TD	1828
SOLVENT	Med
DS	0
NS	64
SWH	4807.692 Hz
FIDRES	0.250036 Hz
AQ	1.9997621 sec
RG	456.1
DM	104.000 usec
TE	300.2 K
TR	3.00 sec
D1	1.00000000 sec
D12	0.00002000 sec
D13	0.00000300 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 usec
P11	4.00 dB
P19	52.29 dB
SFO1	400.1319379 MHz
F2 - Processing parameters	
SF	400.130015 MHz
SI	32768
WDW	GM
SSB	0
LB	-1.61 Hz
GB	0.4
PC	1.00

mwc1101 (epje 1) 298 K

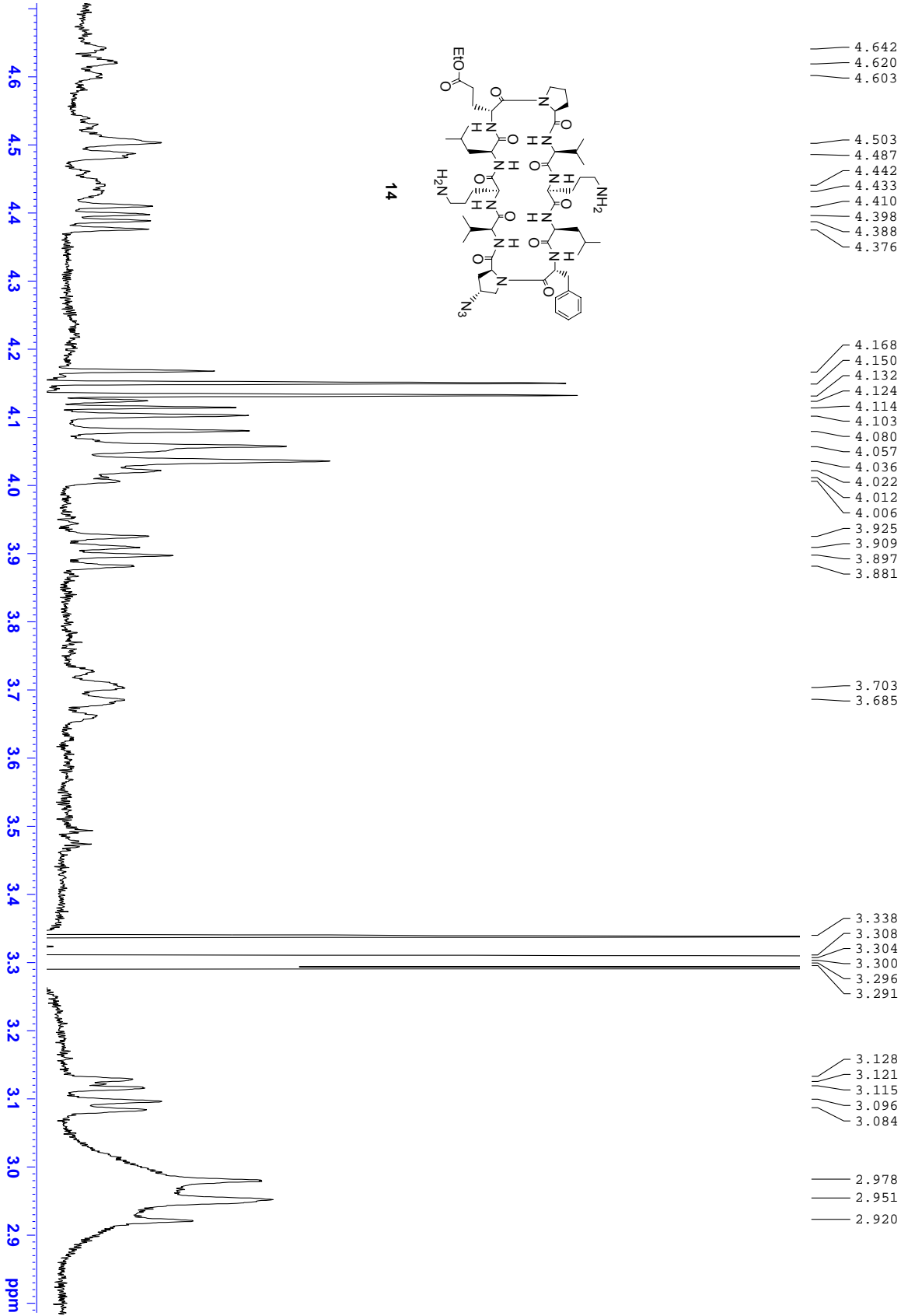


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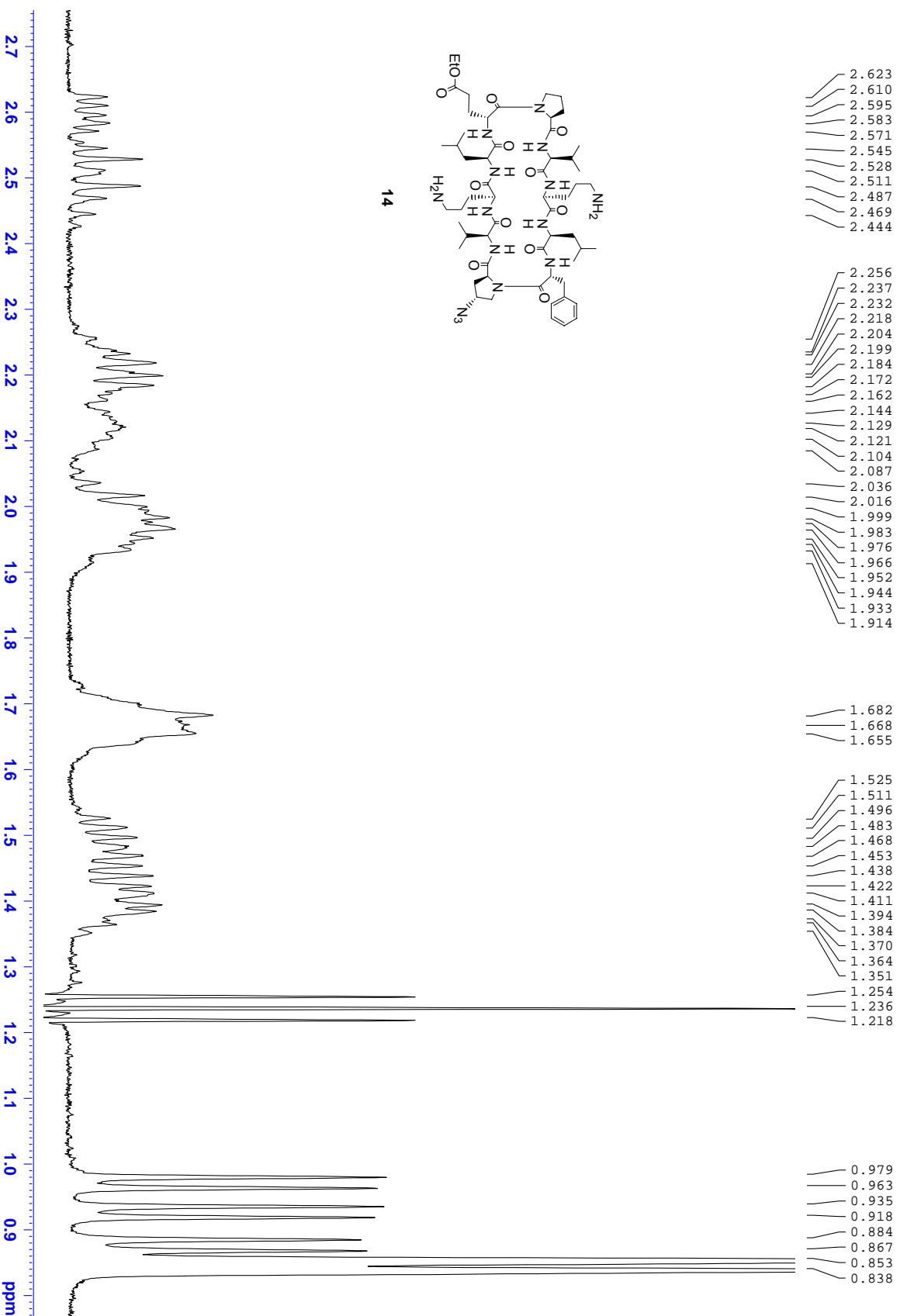
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NAME	glj5042003	
EXPNO	6	
PROCNO	1	
F2 - Acquisition Parameters		
Date_	20030405	
Time	13.21	
Time2	13.21	
Time3	13.21	
PROBHD	5 mm BBI	HS-BB
PULPROG	zgpg	
TD	19238	
SOLVENT	Medd	
DS	0	
NS	64	
SWH	4807.692 Hz	
FIDRES	0.250036 Hz	
AQ	1.9997621 sec	
RG	456.1	
DM	104.000 use	
TE	30.50 use	
TE2	30.50 use	
TD1	1.00000000 sec	
TD2	0.00002000 sec	
TD3	0.00000300 sec	
===== CHANNEL f1 =====		
NUC1	1H	
P1	7.30 use	
P11	4.00 dB	
P19	52.29 dB	
SFO1	400.1319379 MHz	
F2 - Processing parameters		
SI	32768	
WDW	GM	
SSB	0	
LB	-1.61 Hz	
GB	0.4	
PC	1.00	

mwcl101 (epje 1) 298 K



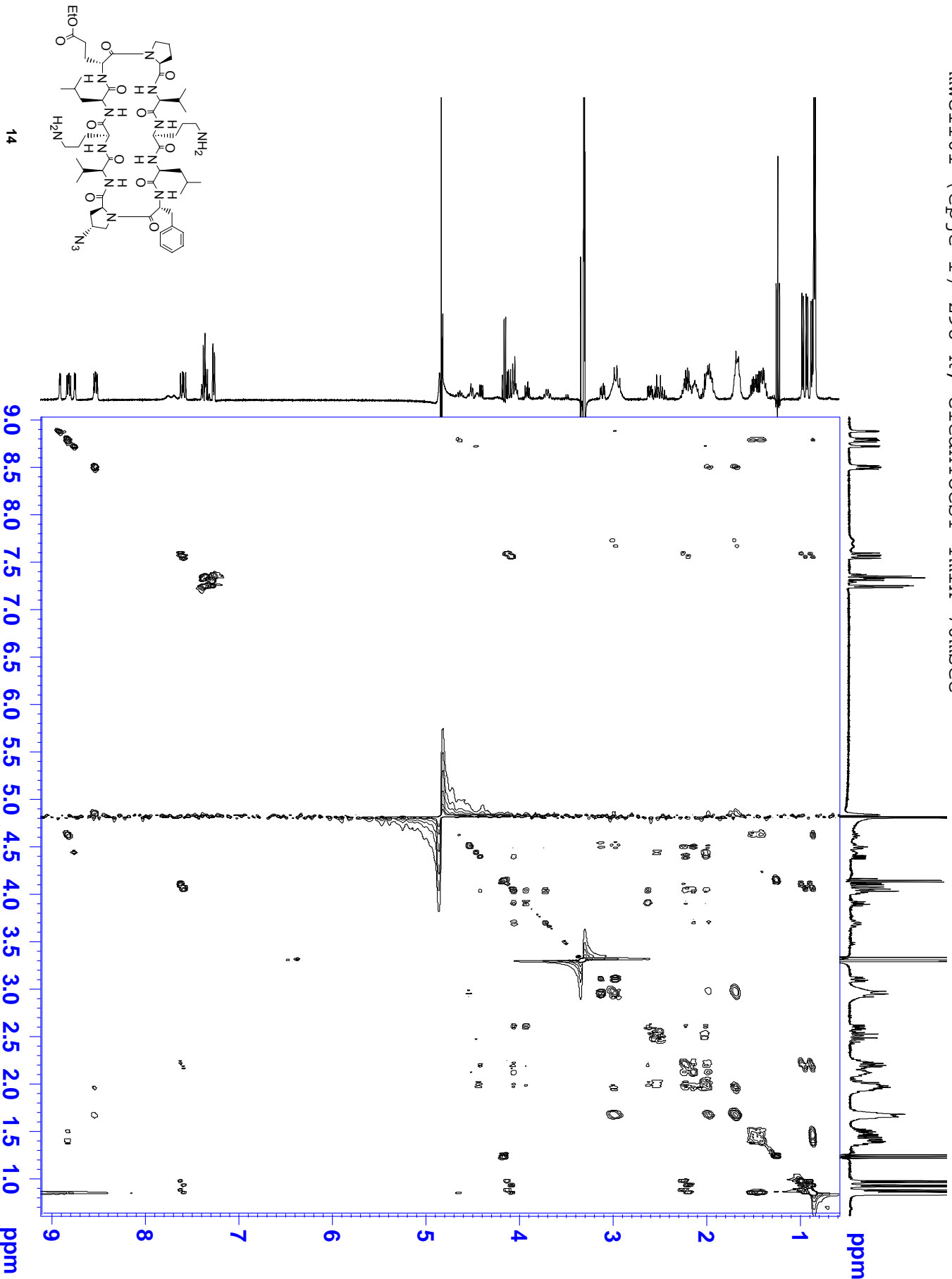
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NAME g1js042003
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters:
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Time 15.21
INSTRUM avance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 13228
FIDRES 0.250036 Hz
RG 456.1
DW 104.000 usi
DE 8.50 usi
TE 300.0 K
D1 1.00000000 sec
d12 0.00002000 sec
d13 0.00000300 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 usi
PL1 4.00 dB
PL2 52.29 dB
PL9 400.131979 MHz
SFO1 400.131979 MHz
F2 - Processing parameters
SI 32768
SF 400.1300115 MHz
WDW GM
SSB 0
LB -1.61 Hz
GB 0.4
PC 1.00

mwc1101 (epje 1) 298 K



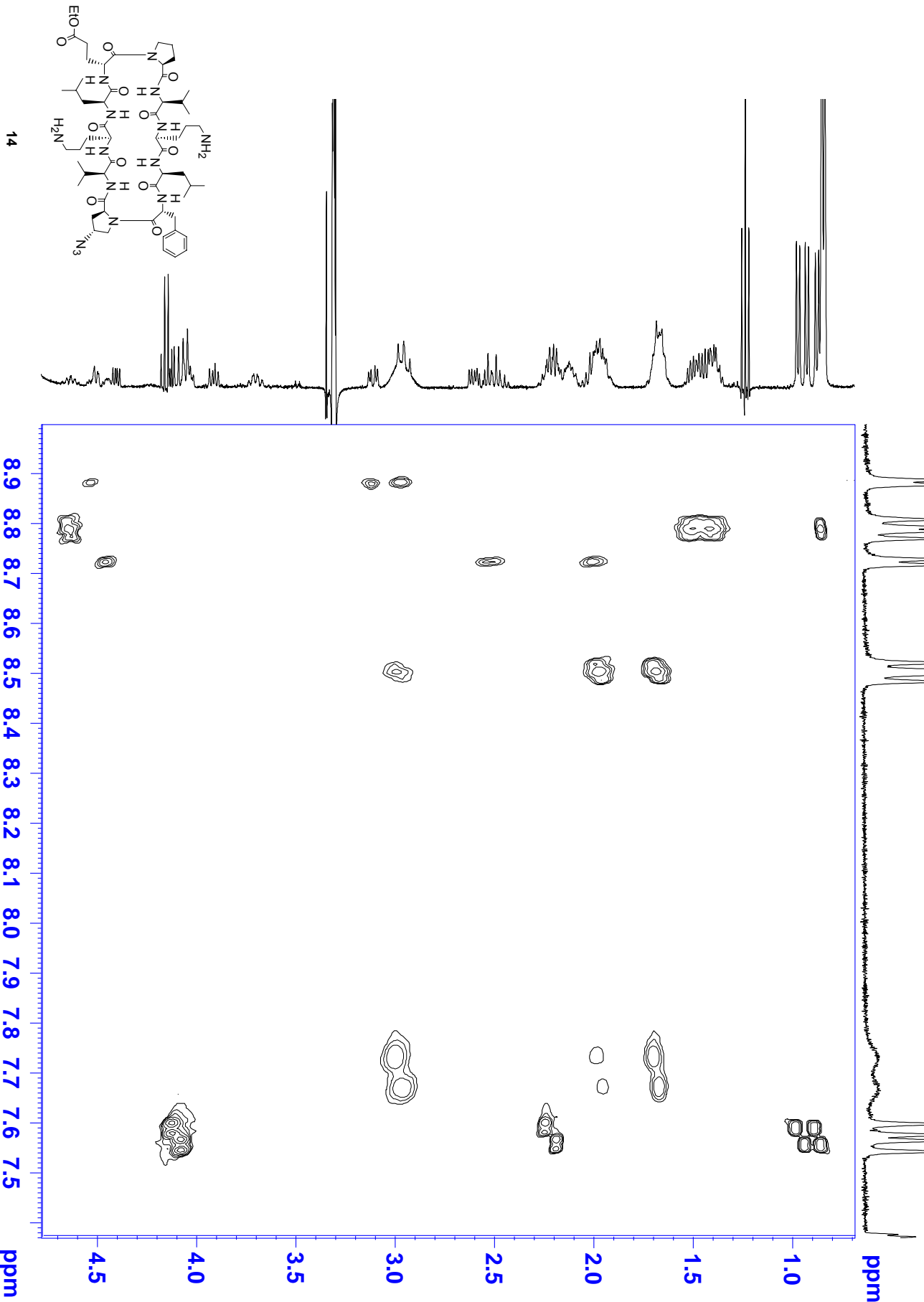
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Date	20030405
Time	13:40
INSTRUM	avaya
PROBHD	5 mm BBI Jr-B8
PULPROG	zgpg
TD	1928
SOLVENT	MeOD
NS	64
DS	0
SWH	4807.692 Hz
FIDRES	0.250036 Hz
AQ	1.997621 sec
RG	456.1
DM	104.000 usec
DE	30.00 usec
TE	300.2 K
TD1	1.00000000 sec
TD2	0.00002000 sec
TD3	0.00000300 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 usec
P11	4.00 dB
P19	52.29 dB
SFO1	400.1319379 MHz
F2 - Processing parameters	
SF	400.130015 MHz
SR	32768
WWM	GM
SSB	0
LB	-1.61 Hz
GB	0.4
PC	1.00

mwcl101 (epje 1) 298 K, cleanTOCSY Tmix=70msec



Current Data Parameters		g13s042003	
NAME	EXNO	PRONO	1
P2 - Acquisition Parameters			
Date_	Time	20030405 15.39	
INSTRUM	PROBHD	5 mm BBI 1H-BB	
PULPROG	SD	zgpg30	
SLVAVMT	DS	CCH3 128	
SWH	FIRRES	3720. 228 Hz 1.816522 Hz	
AQ	RG	0.2753012 sec 2048	
DW	DE	134.400 usec 8.50 usec	
TE		300.0 K	
D0		0.00000300 sec	
D1		1.00000000 sec	
d11		0.03002000 sec	
d12		0.00000000 sec	
d13		0.00000400 sec	
D20		0.00006500 sec	
IN0		0.00026880 sec	
MCNEST		18	
MCNCRF		0.00000000 sec	
STCNT		0.00999999 sec 256	
===== CHANNEL f1 =====			
NUC1		1H	
P1		6.10 usec	
P2		1.10 usec	
P6		25.00 usec	
P7		50.00 usec	
P17		2500.00 usec	
PL9		4.00 dB	
PL10		52.23 dB	
SFO1		400.119375 MHz 16.50 dB	
P1 - Acquisition Parameters			
ND0		1	
SFO1		512 MHz	
PWIDRES		400.119375 Hz	
SW		7.9 296 ppm	
PHMODE		States-tpri	
P2 - Processing parameters			
F2		2048	
MDW		400.130015 MHz	
SSB		OSINE	
LB		2.3 0.00 Hz	
GB		0	
PC		1.00	
P1 - Processing parameters			
SI		1024	
MC2		States-tpri	
SF		400.130006 MHz	
MDW		OSINE	
SSB		2.1	
LB		0.00 Hz	
GB		0	

mwcl101 (epje 1) 298 K, cleanTOCSY Tmix=70msec



Current Data Parameters	
NAME	gljs042003
EXPNO	7
PROCNO	1

```
F2 - Acquisition Parameters
Date_      20030405
Time       15.39
```

INSTRUM	avance400
PROBHD	5 mm BBI 1H-BB
PULPROG	clmlevphyr
TD	2048
SOLVENT	CDCl3
NS	9

NS	9
DS	128
SMH	3720 238 Hz
FIDRES	1.816522 Hz
AQ	0.2753012 sec
RG	2048

DW	134.400	usec
DE	8.50	usec
TE	300.0	K
d0	0.00000300	sec
d1	1.00000000	sec
d11	0.00000000	sec

d11	0.0300000	sec
d12	0.0000200	sec
d13	0.0000400	sec
D20	0.0006500	sec
IN0	0.0002680	sec
L1	18	

```
===== CHANNEL F1 =====
MCREST      0.0000000 sec
MCWRK       0.0099999 sec
STLCNT      256
```

	1H
NOCl	
P1	6.10 usec
P5	16.67 usec
P6	25.00 usec
P7	50.00 usec
P17	2500.00 usec

PL1	4.00 dB
PL9	52.29 dB
PL10	16.50 dB
SFO1	400.1319375 MHz

F1 - Acquisition parameters	
ND0	1
TD	512
SFO1	400.1319 MHz
FIDRES	7.266090 Hz
SW	9.298 mm

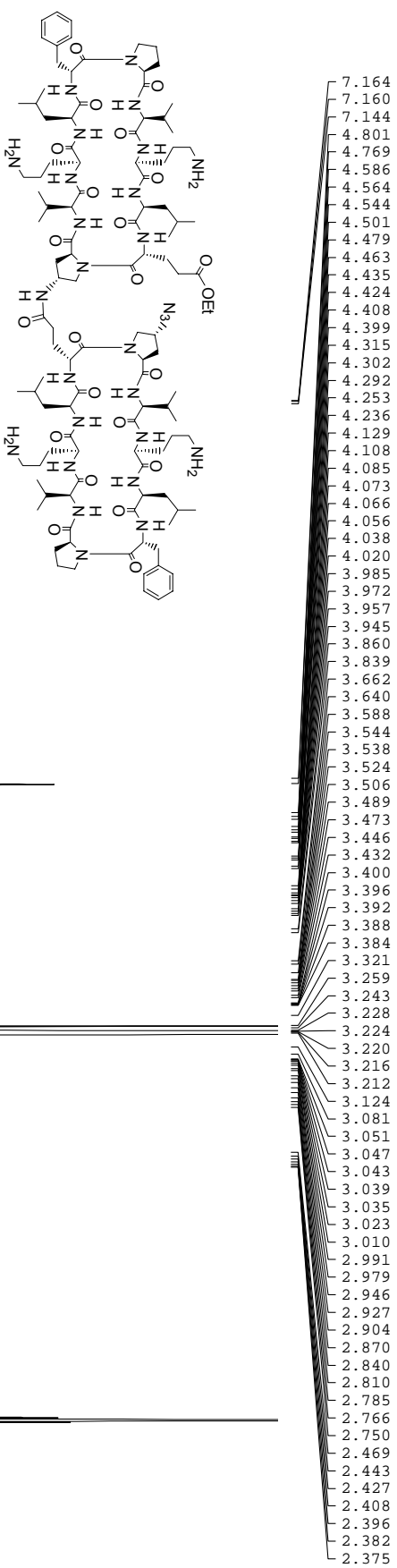
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SI	2048
SF	400.1300115 MHz

WDW	QLINE
SSB	2.3
LB	0.00 Hz
GB	0
PC	1.00

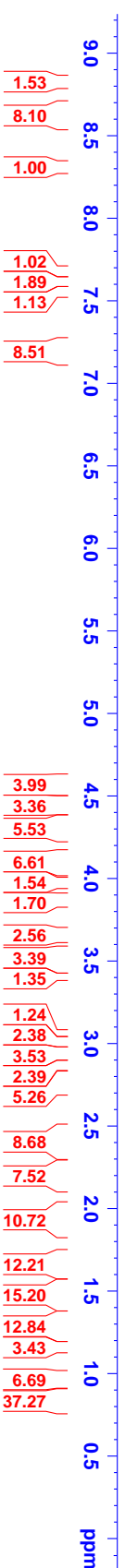
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F1 - Processing parameters
SI      1024
MC2
SF      States-TPI
        400.1300065 MHz
WDW      QSINE
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SSB	2.1
LB	0.00 Hz
GB	0

mwe0806 - dimer N3/Et turn 1H, bbi-av400

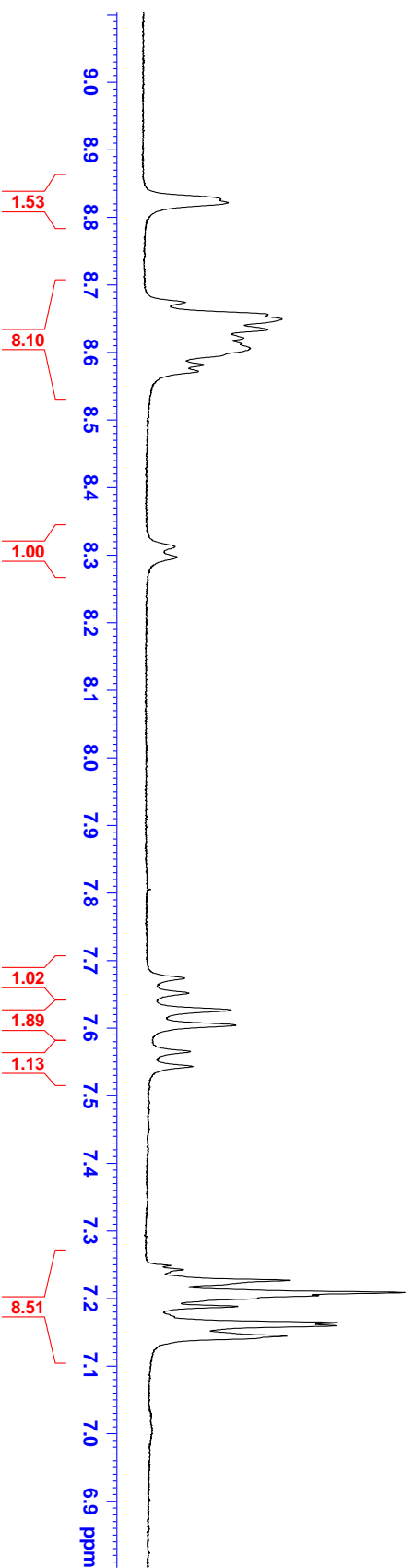
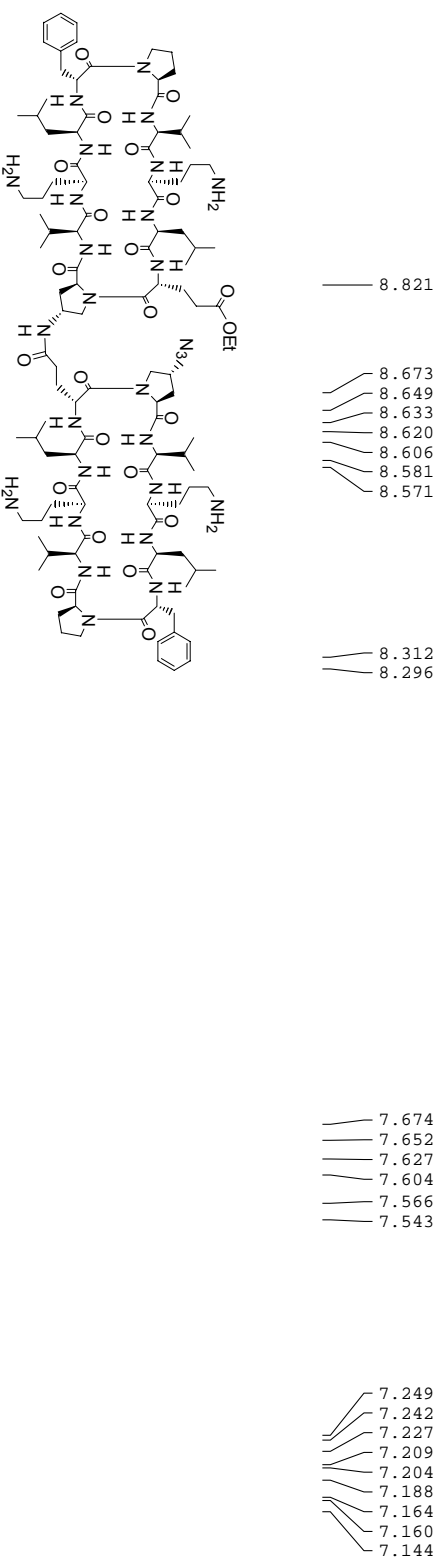


17



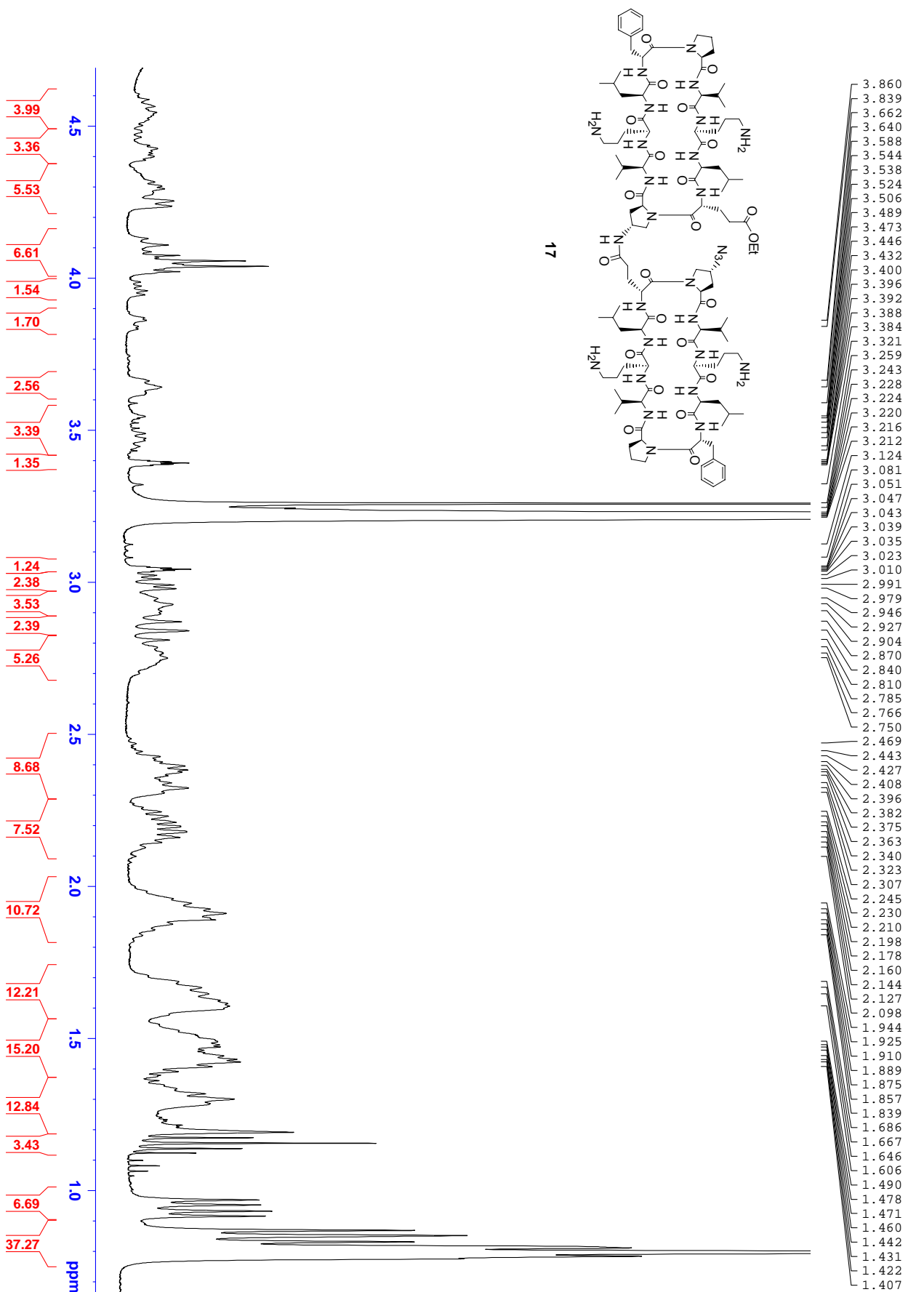
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NAME	GJ5092004
EXNO	1
PRCNO	1
F2 - Acquisition Parameters	
Date-Time	20040915
INSTRUM	10.01
PROBHD	5 mm BI-IB-B
PULPRG	29PR
TD	12228
SOLVENT	H2O
NS	1024
SMH	4807.627 Hz
PIDRFS	0.250036 Hz
AO	1.9997621 sec
RG	256
DW	104.000 usec
DE	8.50 usec
TE	298.0 K
D1	1.00000000 sec
g12	0.00002000 sec
MCESTR	0.00000000 sec
MCWRK	0.01500000 sec
===== CHANNEL f1 =====	
NUCL	1H
P1	7.30 usec
P1A	4.00 dB
P1B	5.00 dB
SFO1	400.1135975 MHz
F2 - Processing parameters	
SI	32768
WDW	400.1300435 MHz
SSB	EM
LB	0.00 Hz
GB	0
PC	1.00

mwe0806 - dimer N3/Et turn 1H, bbi-av400



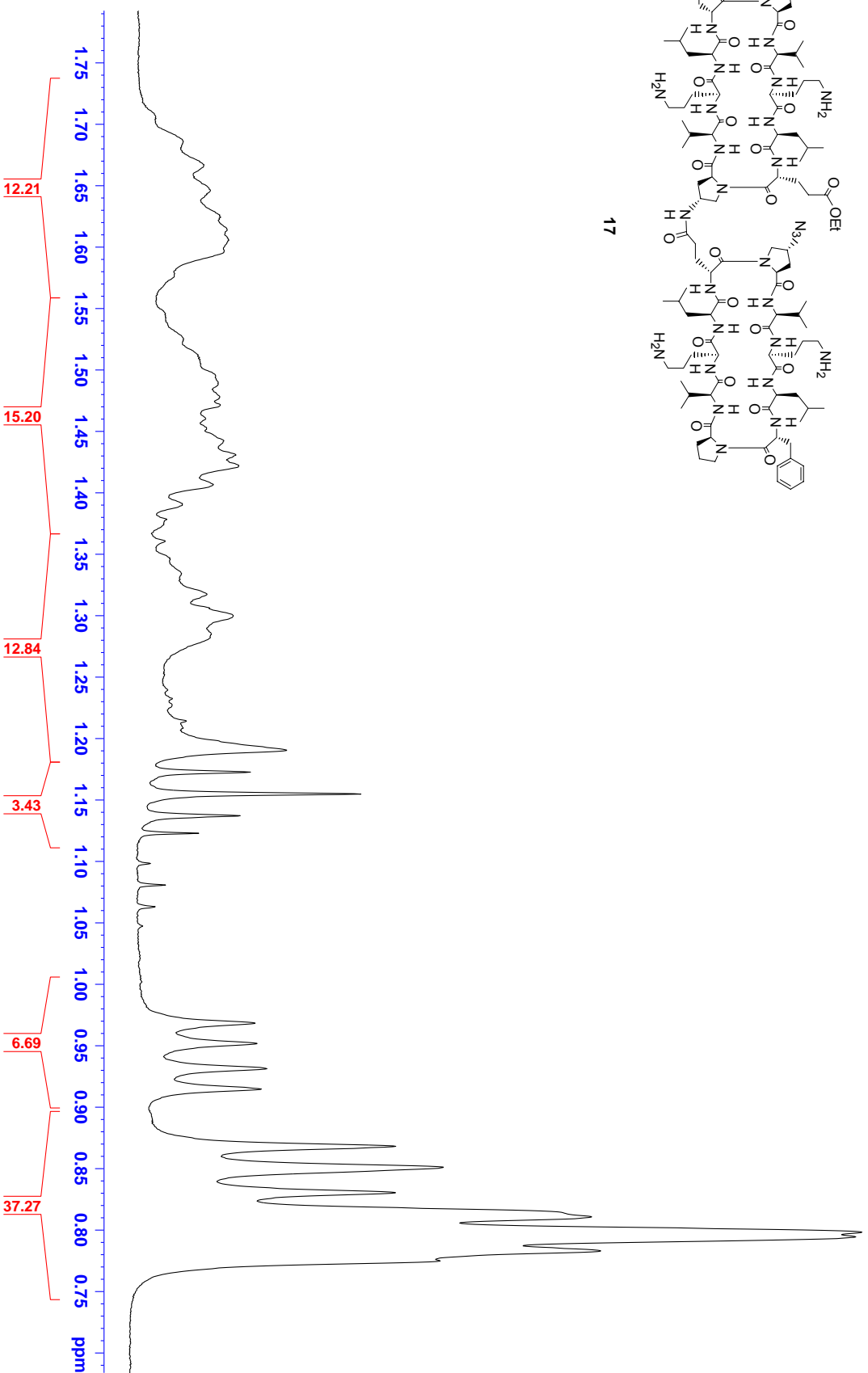
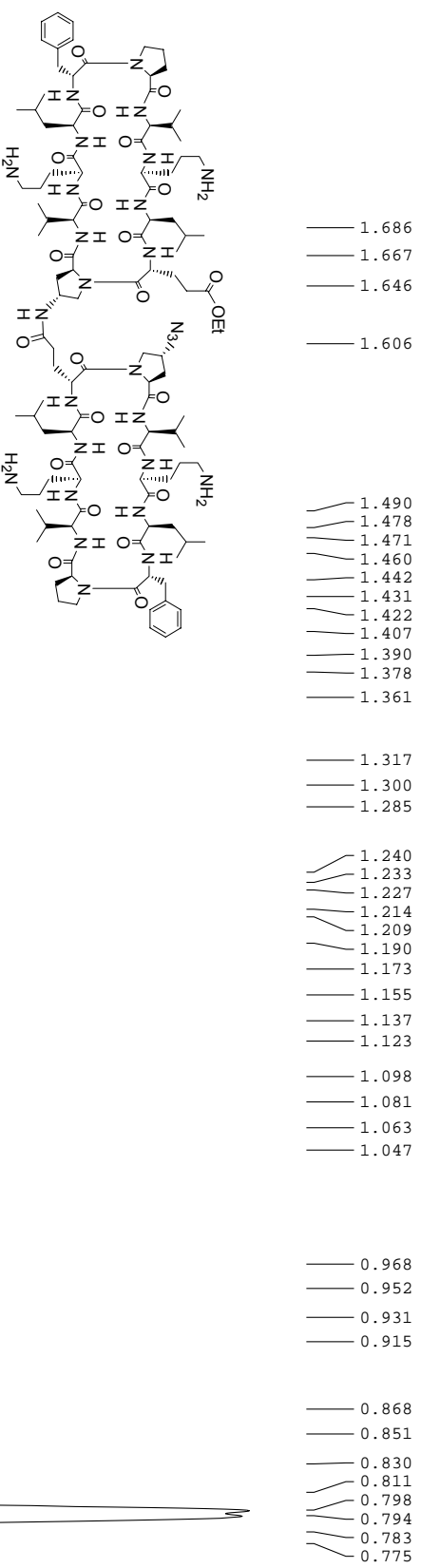
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NAME	GJ3092004
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	20040915
Time	10.01
INSTRUM	avance400
PROBHD	5 mm BBI 1H-4B
PULPROG	zgpg2
TD	19228
SOLVENT	H2O
NS	1024
DS	
SWH	4807.600 Hz
F2	0.250026 Hz
AQ	1.9997621 sec
RG	3256
DW	104.000 usec
DE	8.50 usec
TE	298.0 K
D1	1.00000000 sec
d12	0.00002000 sec
MC12	0.00000000 sec
MEMRK	0.01500000 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 usec
PL1	4.00 dB
P2	58.74 dB
SFO1	400.131975 MHz
F2 - Processing parameters	
SI	400.130045 MHz
SF	373768
MW	EM
LB	0
GB	0.00 Hz
PC	0
GC	1.00

mwe0806 - dimer N3/Et turn 1H, bbi-av400



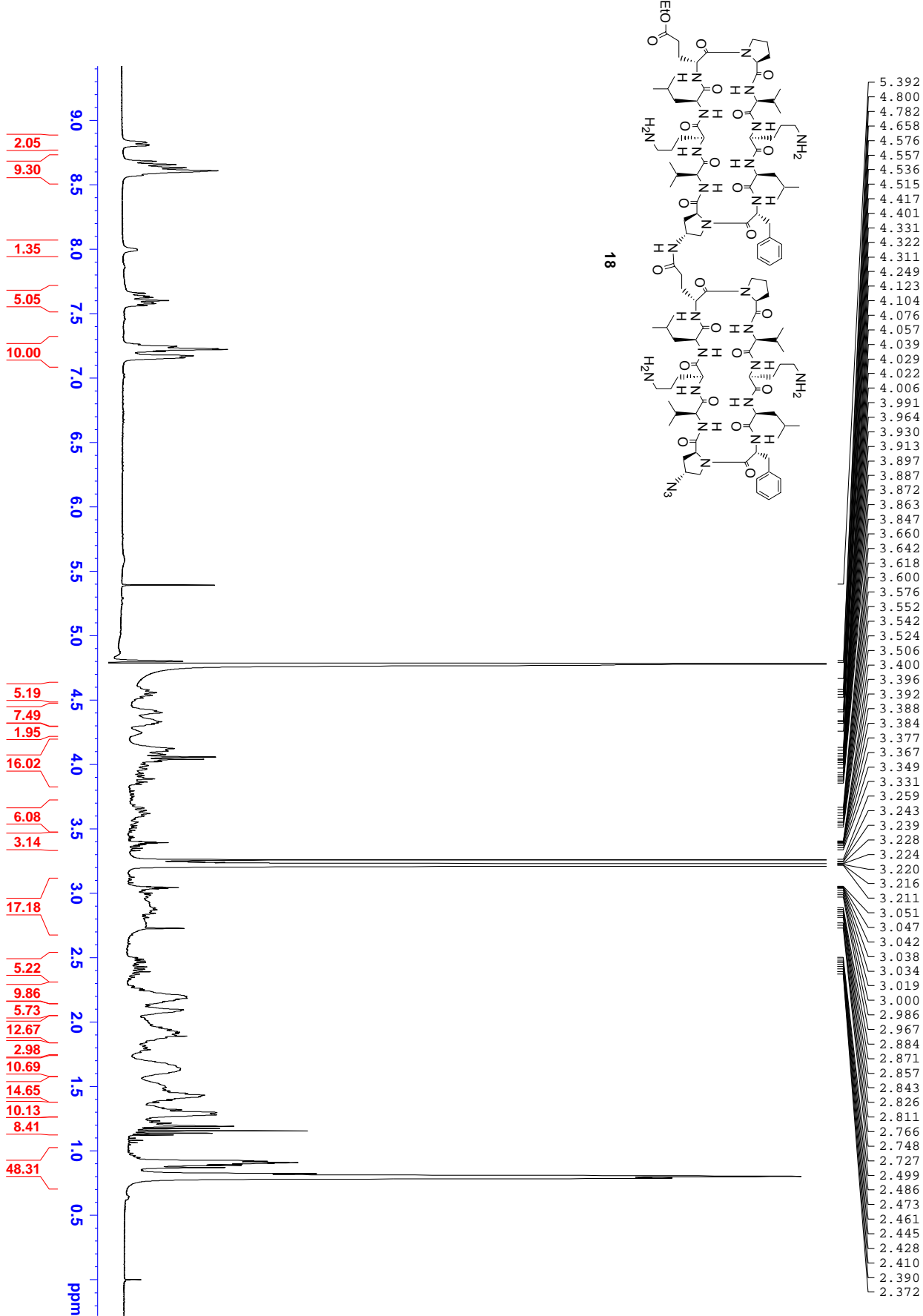
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NAME	Gf5092004
EXNO	1
PRCNO	1
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Time	10.01
INSTRM	avance400
PROBHD	5 mm BBI 1H-BB
PULPROG	zgpg2
TD	19228
NS	H2O
DS	1024
SM	4807.692 Hz
SRH	0.250036 Hz
FIDRES	1.9997621 sec
AO	256
RG	256
DW	104.000 usec
DM	8.50 usec
TE	298.0 K
D1	1.00000000 sec
d12	0.00002000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 usec
PL1	4.00 dB
HD	56.00 Hz
SFO1	400.1319675 MHz
F2 - Processing parameters	
SI	32768
WDW	400.1300435 MHz
SSB	EM
LB	0.00 Hz
GB	0
PC	1.00

mwe0806 - dimer N3/Et turn 1H, bbi-av400



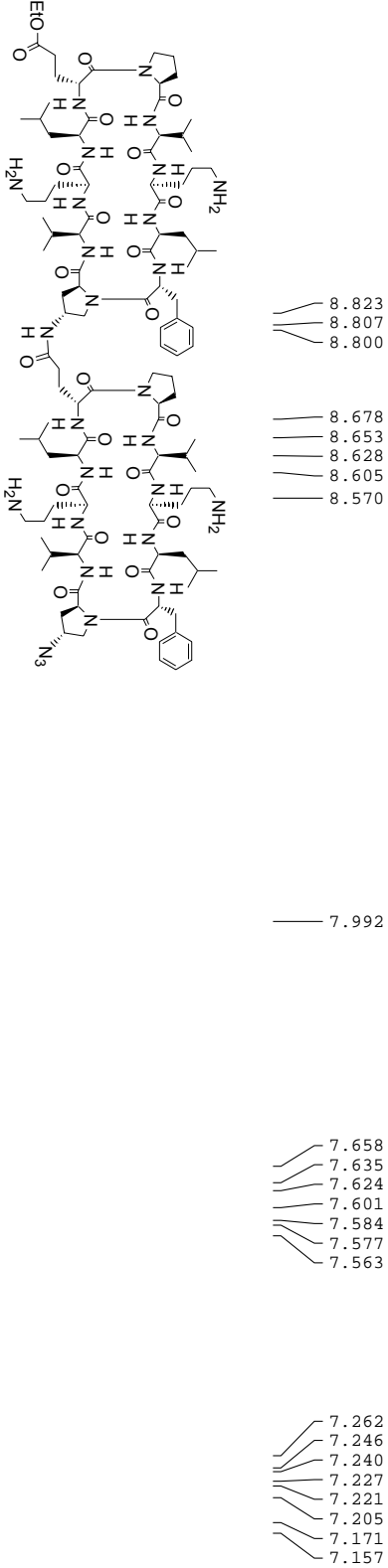
Current Data Parameters	
NAME	G15092004
EXNO	1
PROCN0	1
F2 - Acquisition Parameters	
Date_	20040915
Time	10:01
INSTRUM	avanc400
PROBHD	5 mm BBI 1H-BB
PULPROG	zgpg2
TD	19228
SOLVENT	H2O
NS	1024
DS	0
SWH	4807.692 Hz
F2H1	0.25003 Hz
FINDRES	0.000000 sec
RG	1.9997821 sec
RG	256
DM	104.000 usec
DE	8.50 usec
TE	298.0 K
D1	1.0000000 sec
d12	0.0002000 sec
MCESTRT	0.0000000 sec
MCWRK	0.0150000 sec
===== CHANNEL f1 =====	
NUC01	1H
P1	7.30 usec
PL1	4.00 dB
PL12	58.54 dB
SFO1	400.1319975 MHz
F2 - Processing parameters	
SI	32768
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PB	1.00

mw0801 - dimer N3/Et sheet 1H, bbi-av400

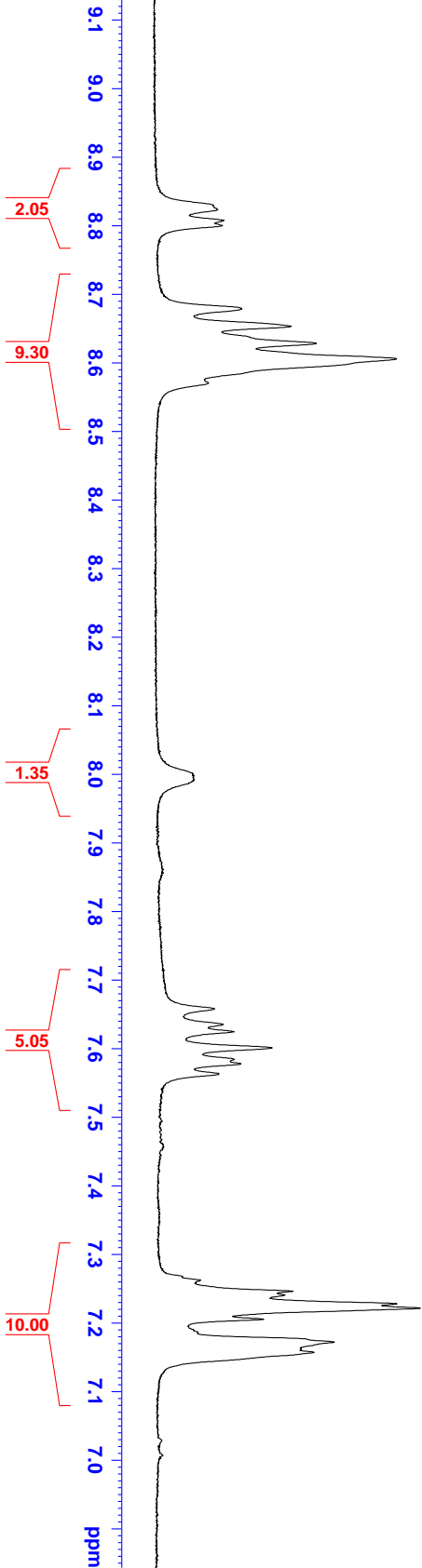


Current Data Parameters
NAME G1j5092004
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040915
Time 13.00
INSTRUM advance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 19228
SOLVENT MeOD
NS 1024
DS 0
SWH 4807.692 Hz
FIDRES 0.250036 Hz
AQ 1.9997621 sec
RG 256
DW 104.000 usec
DE 8.50 usec
TE 283.0 K
D1 1.0000200 sec
d12 0.0000200 sec
MCOREST 0.0000000 sec
MCWRX 0.0150000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 usec
PL1 4.00 dB
PL9 60.89 dB
SF01 400.1319575 MHz
F2 - Processing parameters
SI 400.1300436 MHz
SF 32768
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

mwc0801 - dimer N3/Et sheet 1H, bbi-av400

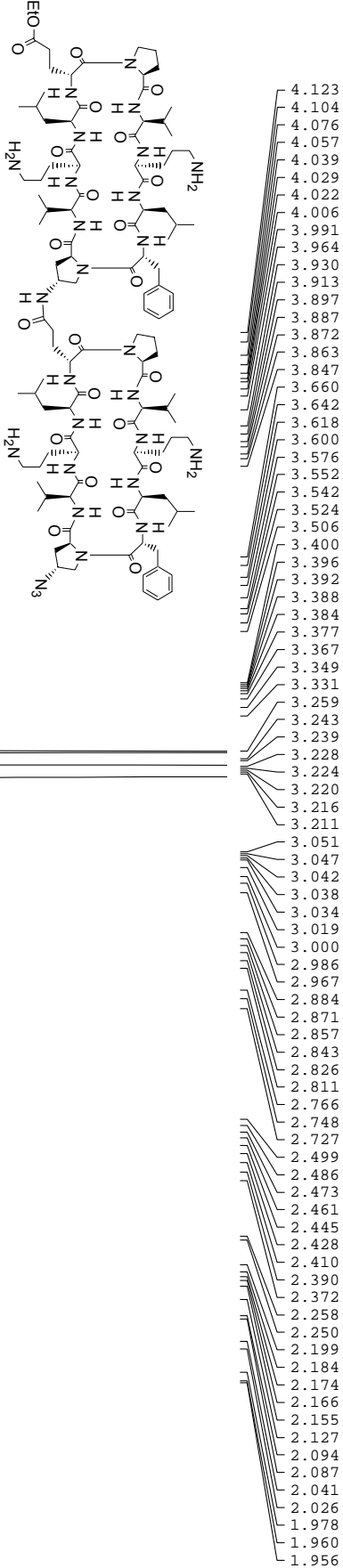


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Current Data Parameters	
NAME	GJ5092004
EXPNO	4
PROCNO	1
F2 - Acquisition Parameters	
Date_	20040915
Time	13.00
INSTRUM	avance400
PROBHD	5 mm BBI 1H-BB
PULPROG	zgpr
TD	19228
SOLVENT	MeOD
NS	1024
DS	0
SWH	4807.692 Hz
FIDRES	0.250036 Hz
AQ	1.9997621 sec
RG	256
DW	104.000 use
DE	8.50 use
TE	298.0 K
DE	1.0000000 sec
DE	0.0000000 sec
MCREST	0.0000000 sec
MCRMK	0.0150000 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 use
PL1	4.00 dB
PL9	60.89 dB
SFO1	400.1319575 MHz
F2 - Processing parameters	
SI	32768
SF	400.1300436 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

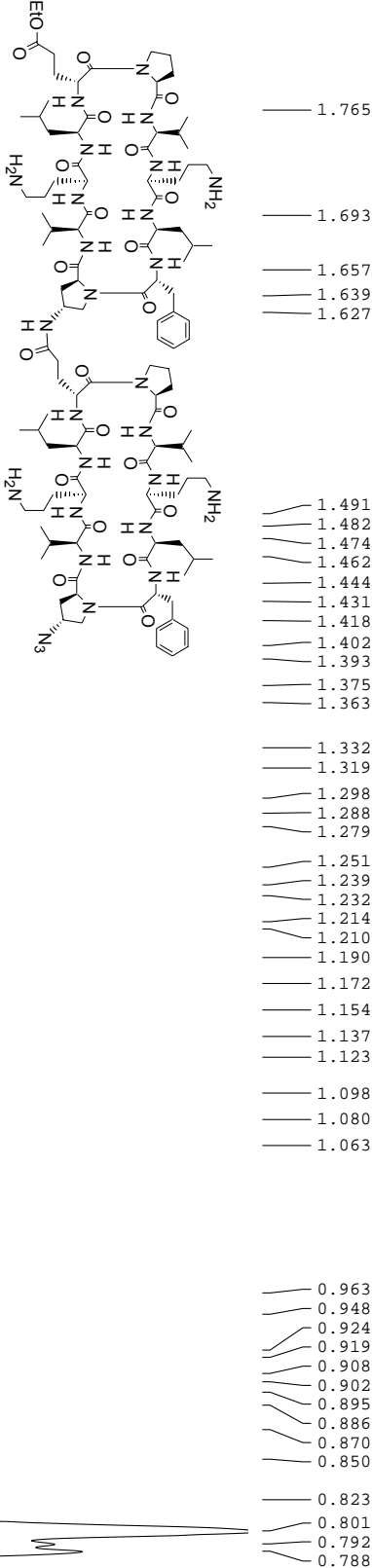
mwc0801 - dimer N3/Et sheet 1H, bbi-av400



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Current Data Parameters
NAME G1j5032004
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040915
Time 13.00
INSTRUM advance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 19228
FIDRES 1.9997621 sec
RG 256
DW 104.000 usec
DE 28.50 usec
TE 300.2 K
D1 1.00002000 sec
d12 0.00002000 sec
MCOREST 0.00000000 sec
MCWRX 0.01500000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 usec
PL1 4.00 dB
PL9 60.89 dB
SF01 400.1319575 MHz
F2 - Processing parameters
SI 400.1300436 MHz
SF 400.1300436 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

mwc0801 - dimer N3/Et sheet 1H, bbi-av400

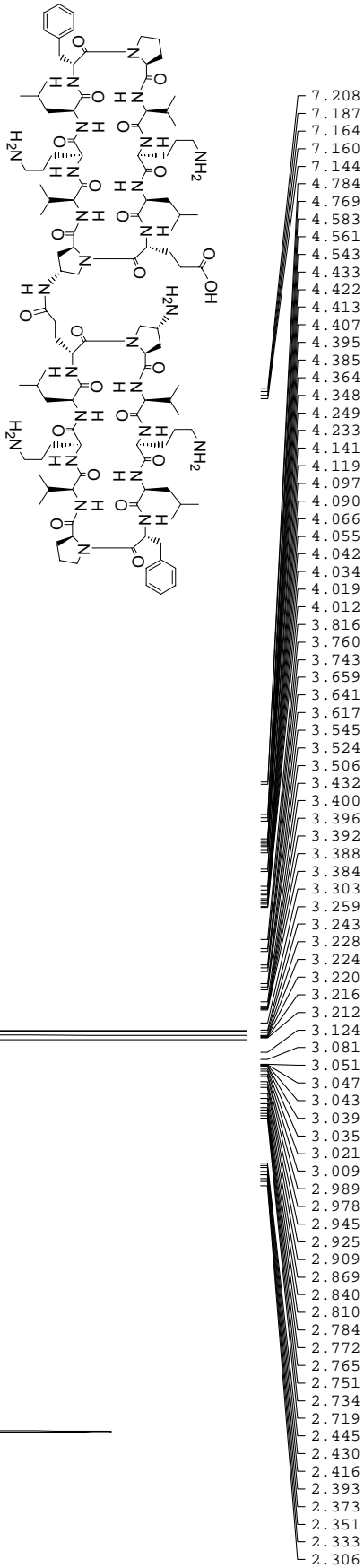


18

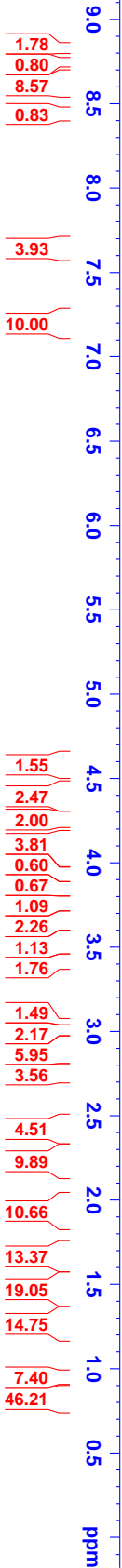


Current Data Parameters
NAME GJ5092004
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040915
Time 13.00
INSTRUM avance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 19228
FIDRES 0.250036 Hz
AQ 1.9997621 sec
RG 286
DE 104.000 usec
DQ 8.50 usec
DE 398.0 K
DI 1.0000000 sec
d12 0.0000200 sec
MCREST 0.0000000 sec
MCWRK 0.01500000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 usec
PL1 4.00 dB
PL9 60.89 dB
SFO1 400.1319575 MHz
F2 - Processing parameters
SI 32768
SF 400.1300436 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

mwel501 - dimer NH2/COOH turn 1H, bbi-av400

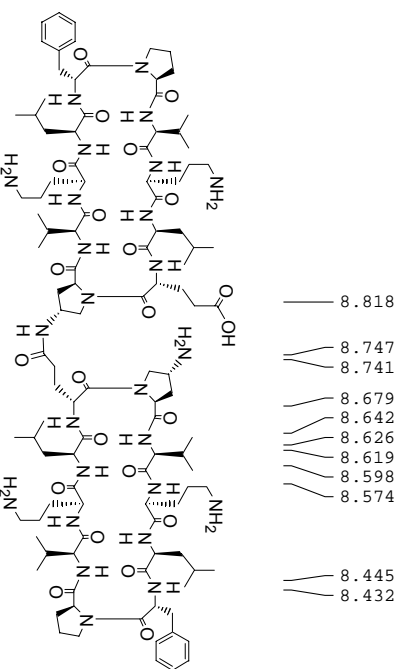


21

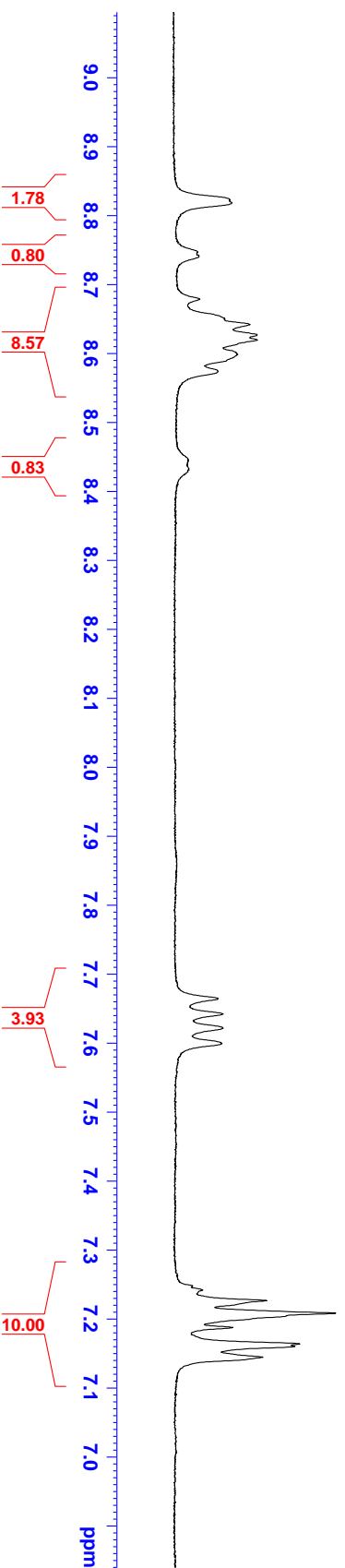


Current Data Parameters
NAME G1j502004
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040915
Time 11.43
INSTRUM avance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 19228
SOLVENT MeOD
NS 1024
DS 0
SWH 4807.692 Hz
FIDRES 0.250036 Hz
AQ 1.997621 sec
RG 256
DW 104.000 usec
DE 8.50 usec
TE 300.2 K
D1 1.0000200 sec
d12 0.0000200 sec
MCREST 0.0000000 sec
MCMRK 0.0150000 sec
==== CHANNEL f1 =====
NUC1 1H
P1 7.30 usec
PL1 4.00 dB
PL9 58.54 dB
SFO1 400.1319575 MHz
F2 - Processing parameters
SI 32768
SF 400.1300435 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

mw1501 - dimer NH2/COOH turn 1H, bbi-av400



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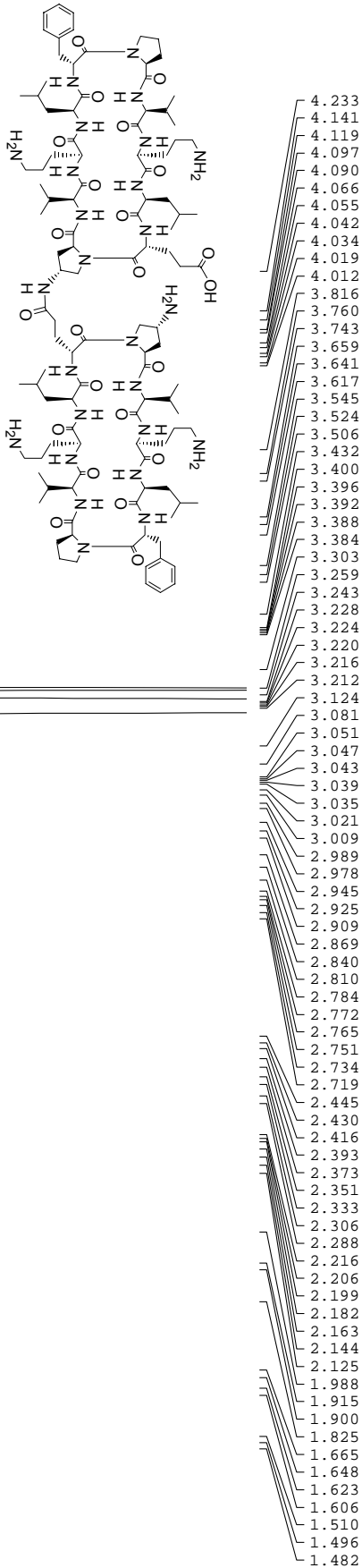


~~7.665~~
~~7.642~~
~~7.621~~
~~7.599~~

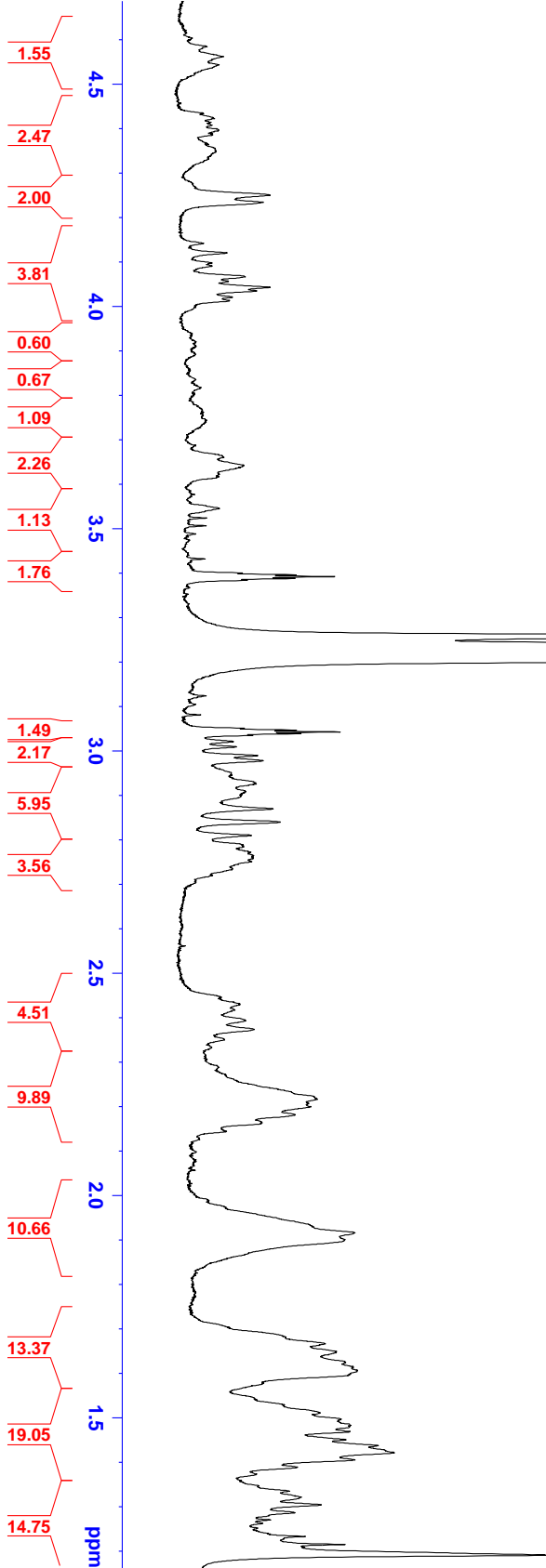
7.247
7.242
7.226
7.208
7.187
7.164
7.160
7.144

Current Data Parameters	
NAME	G3092004
EXPNO	3
PROCNO	1
F2 - Acquisition Parameters	
Date_	20040915
Time	11.43
INSTRUM	advantc400
PROBHD	5 mm BBI 1H-4B0
PULPROG	zgpg
TD	19228
SOLVENT	MeOD
NS	1024
DS	0
SWH	4897.680 Hz
F2	0.950023 Hz
AQ	1.9997621 sec
RG	325
DE	104.000 usec
DD	8.50 usec
TE	298.0 K
D1	1.00000000 sec
d12	0.00020000 sec
MC1RST	0.00000000 sec
MEMRK	0.01500000 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 usec
PL1	4.00 dB
P2	58.54 dB
SFO1	400.131975 MHz
F2 - Processing parameters	
F2	400.130045 MHz
SF	37978
WOW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

mwel1501 - dimer NH2/COOH turn 1H, bbi-av400

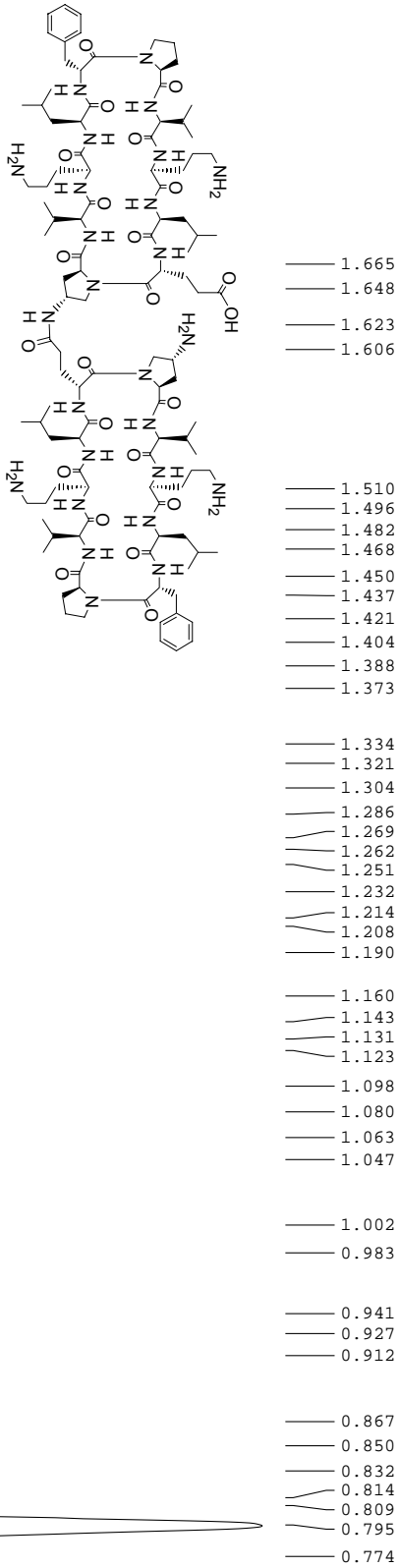


21

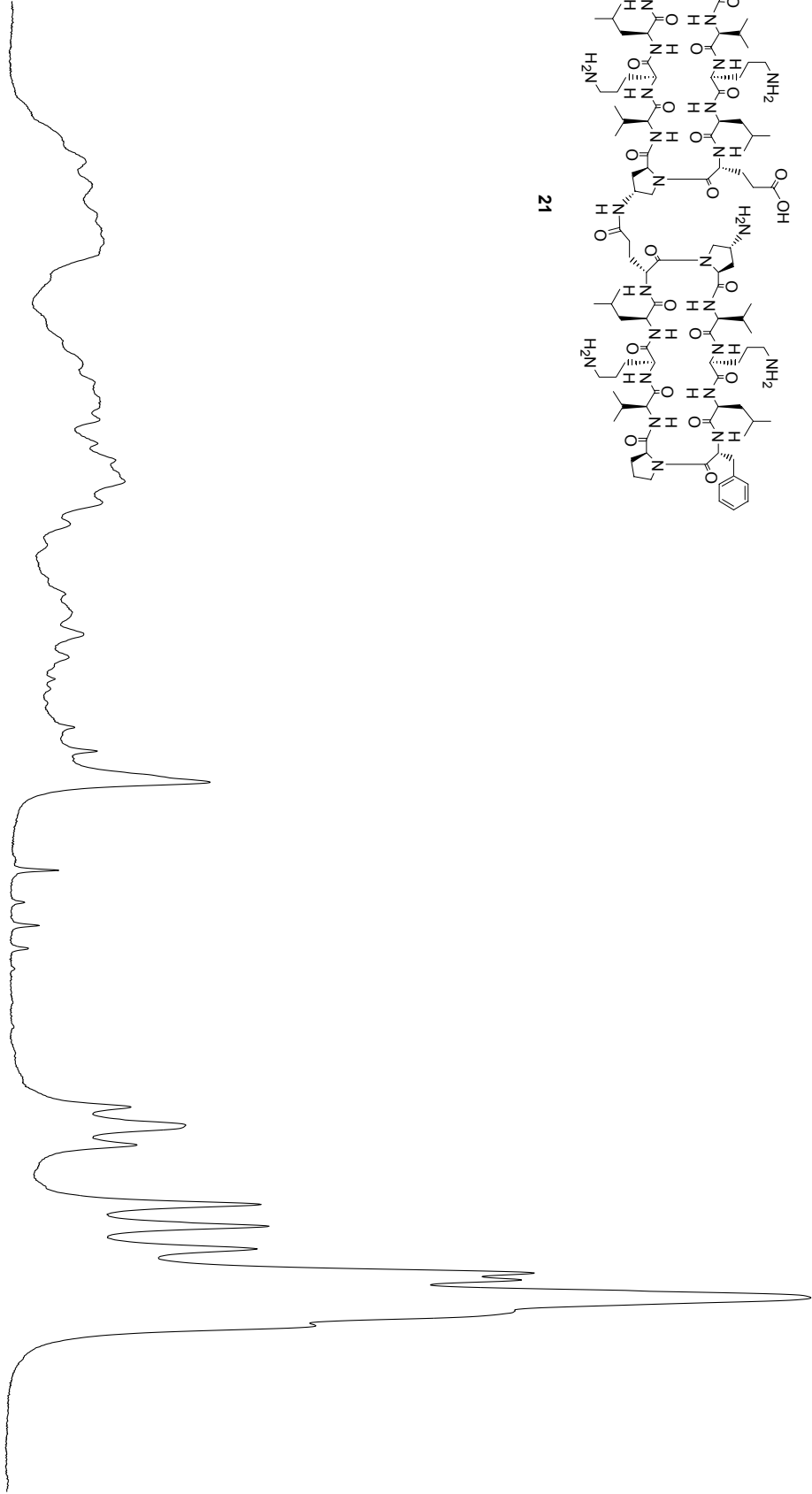


Current Data Parameters
NAME G1j5092004
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040915
Time 11.43
INSTRUM avance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 19228
SOLVENT MeOD
NS 1024
DS 0
SWH 4807.692 Hz
FIDRES 0.25036 Hz
AQ 1.9997621 sec
RG 256
DW 104.000 usec
DE 8.50 usec
TE 300.2 K
D1 1.00002000 sec
d12 0.00002000 sec
MCOREST 0.00000000 sec
MCWRX 0.01500000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 usec
PL1 4.00 dB
PL9 58.54 dB
SF01 400.1319575 MHz
F2 - Processing parameters
SI 32768
SF 400.1300435 MHz
WDW EM
SSB 0
LB 0
GB 0
PC 1.00

mwel501 - dimer NH2/COOH turn 1H, bbi-av400

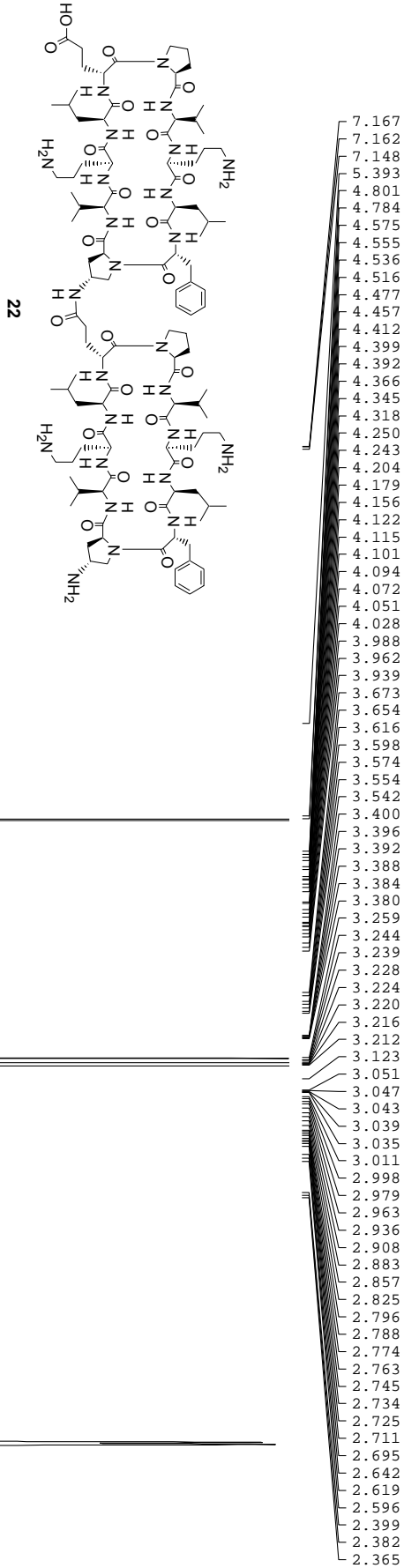


21



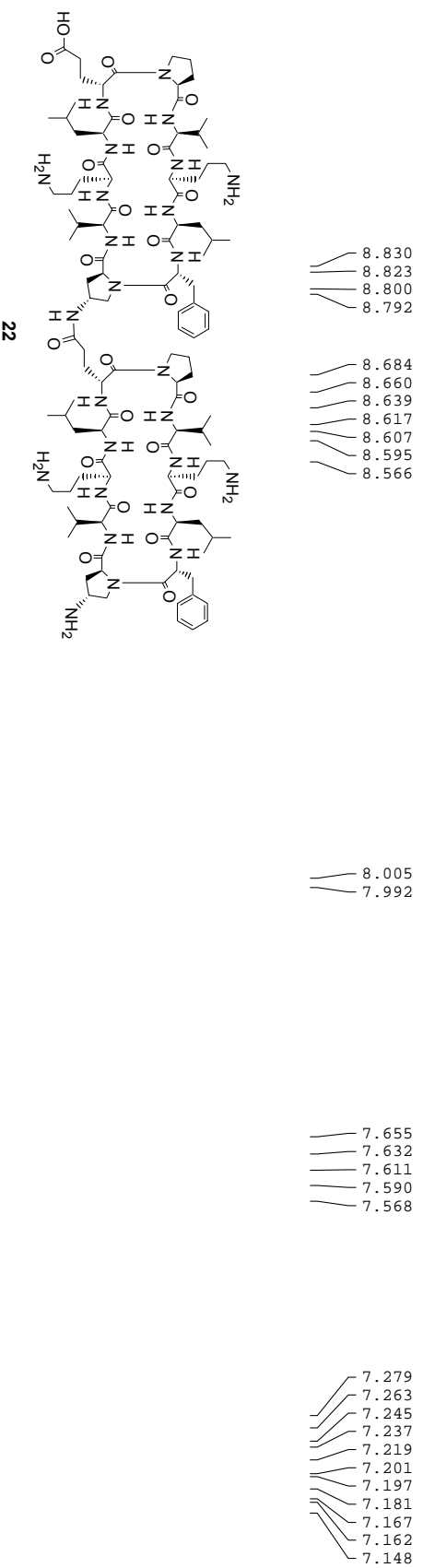
Current Data Parameters
NAME GJ5092004
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040915
Time_ 11.43
INSTRUM avance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpr
TD 19228
SOLVENT MeOD
NS 1024
DS 0
SWH 4807.692 Hz
FIDRES 0.250036 Hz
AQ 1.9997621 sec
RG 286
DW 104.000 usec
DE 8.50 usec
TE 298.0 K
D1 1.0000000 sec
d12 0.0000200 sec
MCREST 0.0000000 sec
MCWRK 0.01500000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 usec
PL1 4.00 dB
PL9 58.54 dB
SFO1 400.1319575 MHz
F2 - Processing parameters
SI 32768
SF 400.1300435 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

mwcl501 - dimer NH2/COOH sheet 1H, bbi-av400



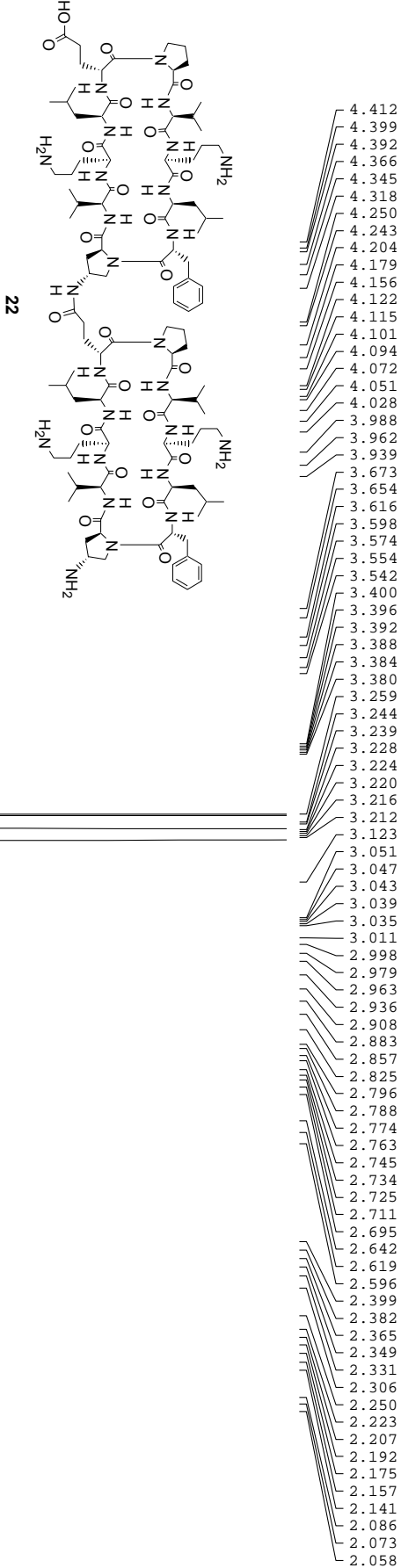
Current Data Parameters
NAME G1j502004
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040915
Time 14.09
INSTRUM avance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 19228
SOLVENT MeOD
NS 1024
DS 0
SWH 4807.692 Hz
FIDRES 0.25036 Hz
AQ 1.9997621 sec
RG 256
DW 104.000 usec
DE 8.50 usec
TE 283.0 K
D2 1.00002000 sec
d12 0.00002000 sec
MCOREST 0.00000000 sec
MCMRK 0.01500000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 usec
PL1 4.00 dB
PL9 60.89 dB
SF01 400.1319579 MHz
F2 - Processing parameters
SI 400.1300433 MHz
SF 32768
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

mmw1501 - dimer NH₂/COOH sheet 1H, bbi-av400



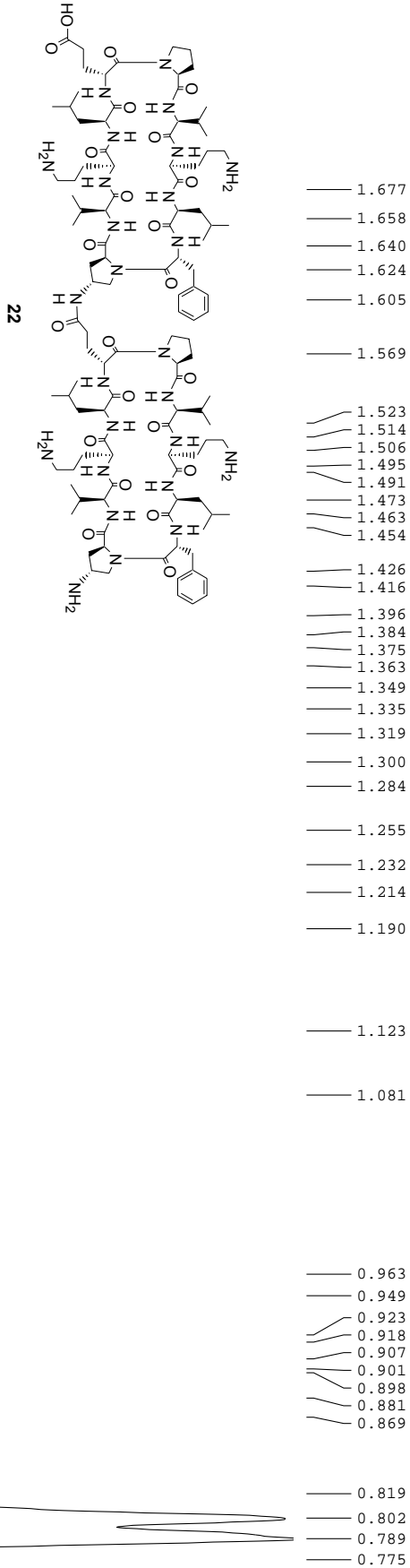
Current Data Parameters	
NAME	G16092004
EXNO	5
PRONO	1
F2 - Acquisition Parameters	
Time	20040915
Time	14.09
INSTRUM	avance400
PROBHD	5 mm BBI-1H-BB
PULPROG	zgpg
TD	19228
SOLVENT	MEOd
NS	1024
DS	0
SWH	4807.692 Hz
F2H1	0.250033
NUC1P1	1.9997623 sec
RG	256
DE	104.000 use
DM	8.50 use
TE	298.0 K
D1	1.00000000 sec
d12	0.00002000 sec
MCHEST	0.00000000 sec
MCWRR	0.0150000 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 use
P1L	4.00 dB
P1B	60.89 dB
SFO1	400.1319579 MHz
F2 - Processing Parameters	
SI	32768
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PB	1.00

mwcl501 - dimer NH2/COOH sheet 1H, bbi-av400

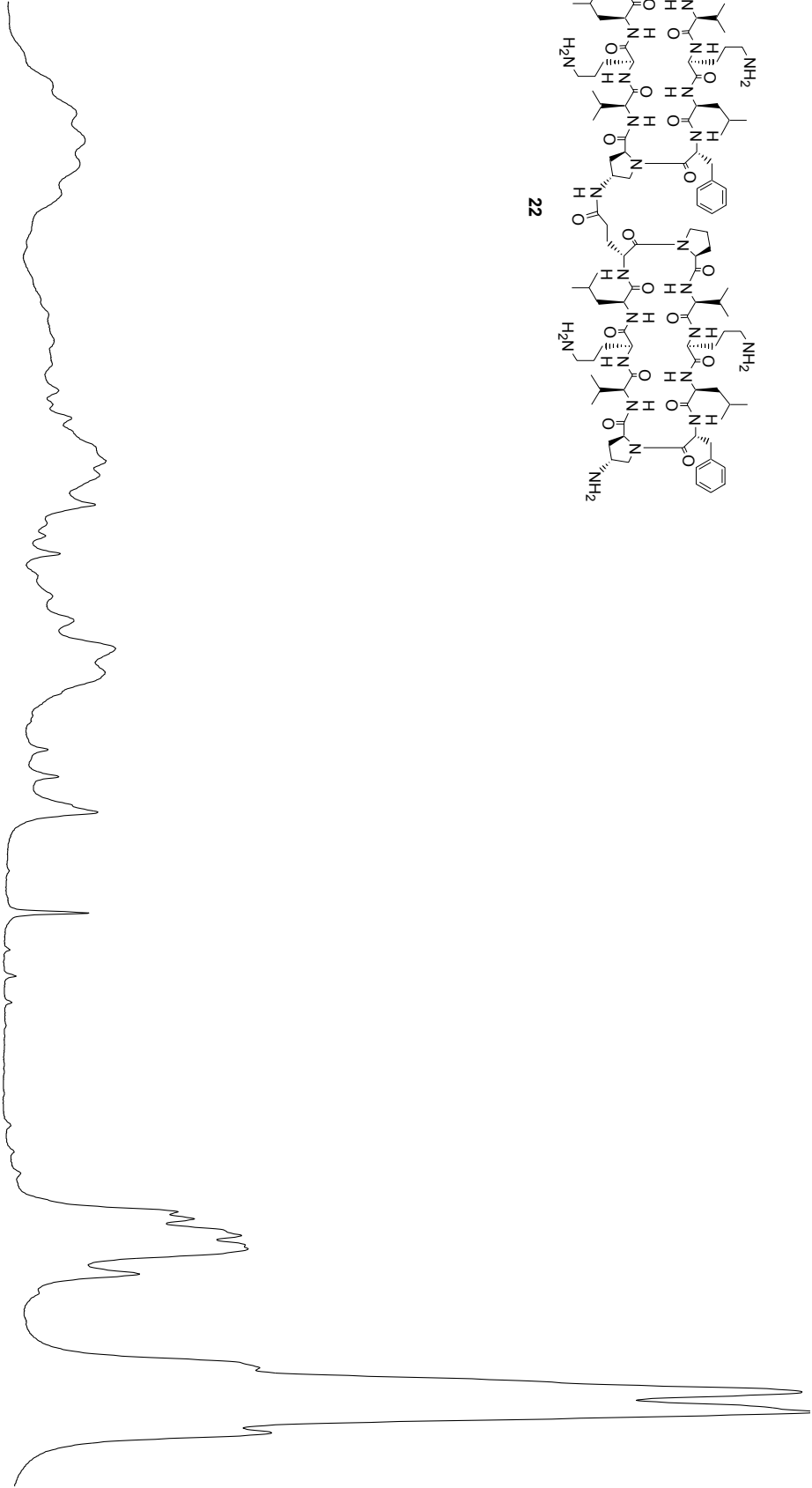


Current Data Parameters
NAME G1j502004
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040915
Time 14.09
INSTRUM advance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 19228
SOLVENT MeOD
NS 1024
DS 0
SWH 4807.692 Hz
FIDRES 0.250036 Hz
AQ 1.9997621 sec
RG 256
DW 104.000 usec
DE 8.50 usec
TE 283.0 K
D1 1.00000000 sec
d12 0.00002000 sec
MCREST 0.00000000 sec
MCWRX 0.01500000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 usec
PL1 4.00 dB
PL9 60.89 dB
SF01 400.1319579 MHz
F2 - Processing parameters
SI 400.1300433 MHz
SF 400.1300433 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

mwcl501 - dimer NH2/COOH sheet 1H, bbi-av400

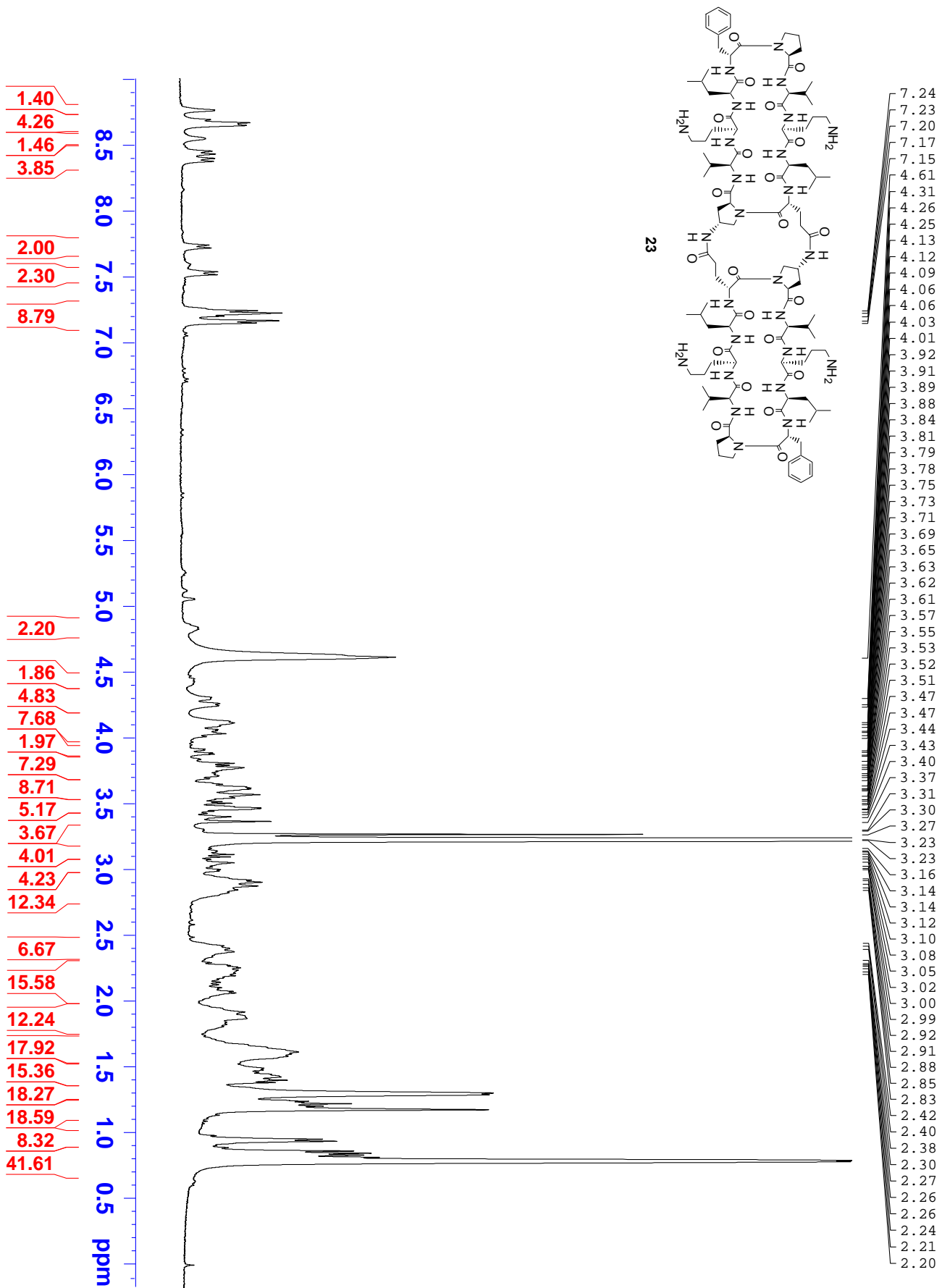


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Current Data Parameters
NAME G:\j5092004
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040915
Time_ 14.09
INSTRUM avance400
PROBHD 5 mm BBI 1H-BB
PULPROG zgpg
TD 19228
FIDRES 0.250036 Hz
RG 104.000 use
DE 8.50 use
DS 0
SOLVENT MeOD
NS 1024
SWH 4807.692 Hz
FIDRES 0.250036 Hz
AQ 1.9997621 sec
RG 286
DW 104.000 use
DE 8.50 use
TE 298.0 K
D1 1.00000000 sec
d12 0.00002000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 7.30 use
PL1 4.00 dB
PL9 60.89 dB
SFO1 400.1319579 MHz
F2 - Processing parameters
SI 32768
SF 400.1300433 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

dimer over turn 1h nmr av-400 ATM-bbiggs probe

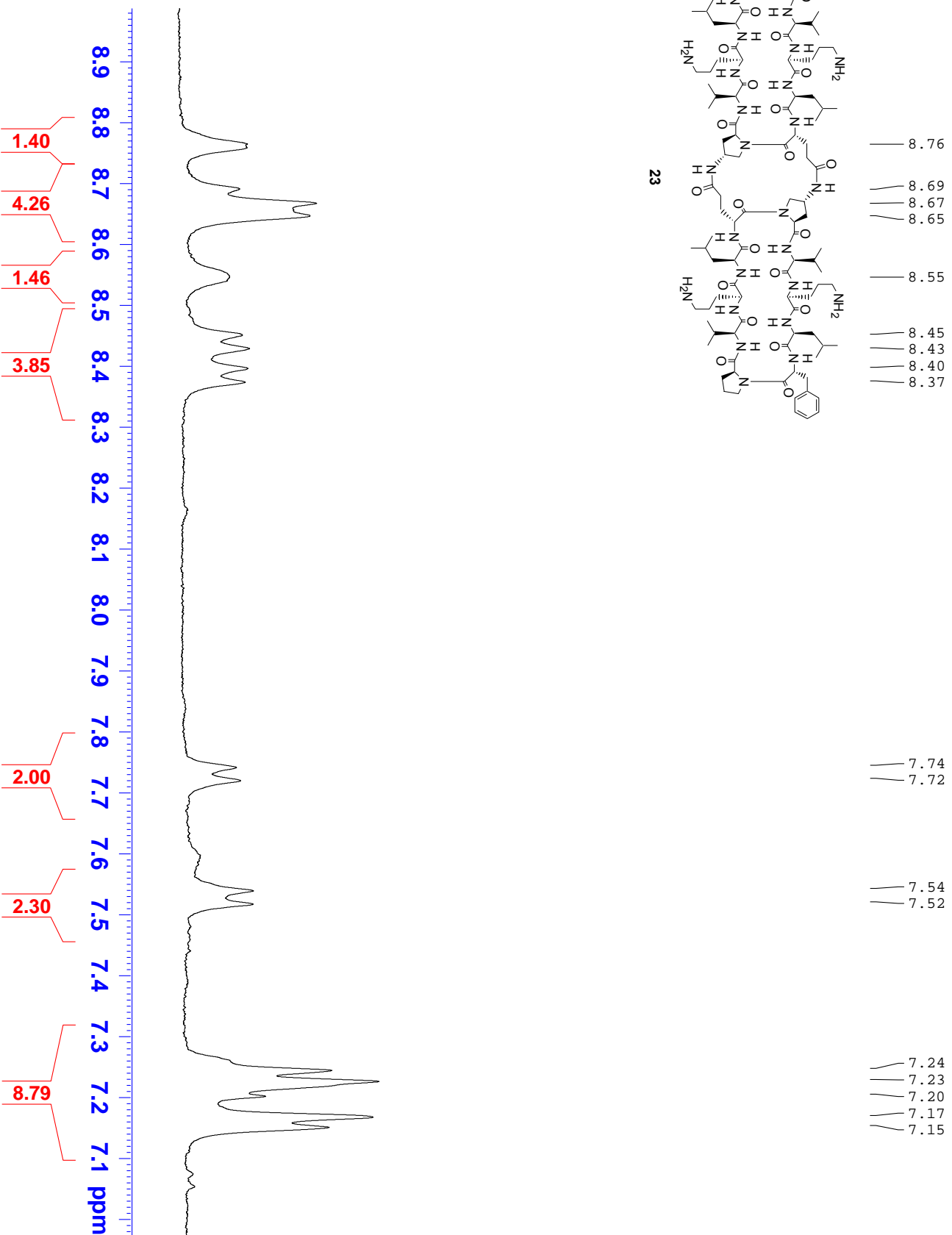
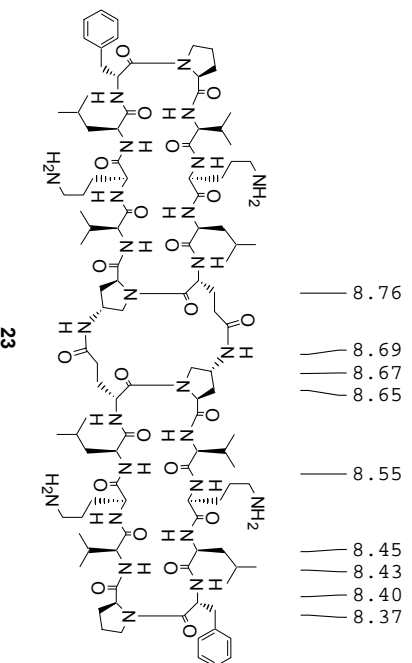


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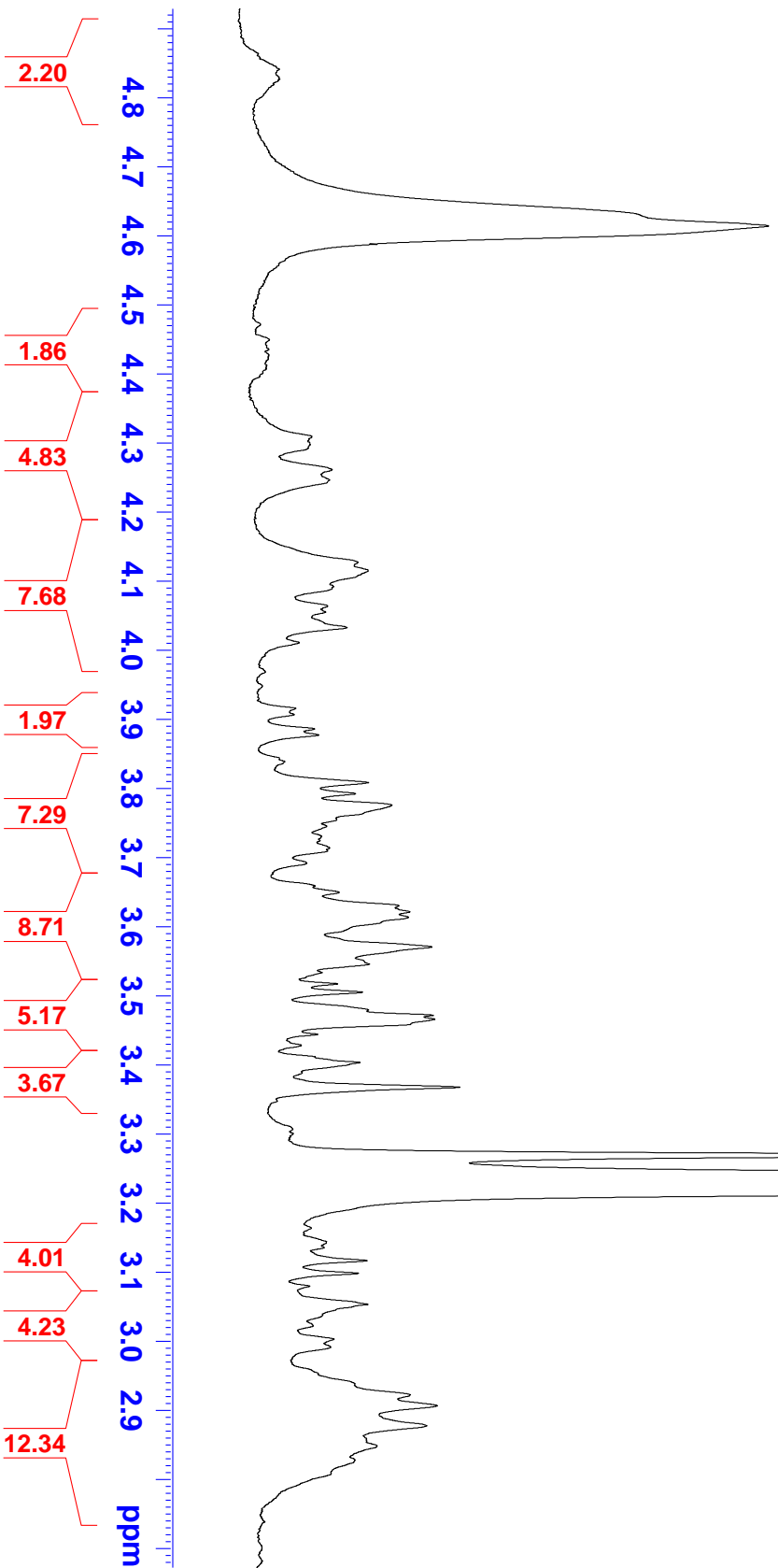
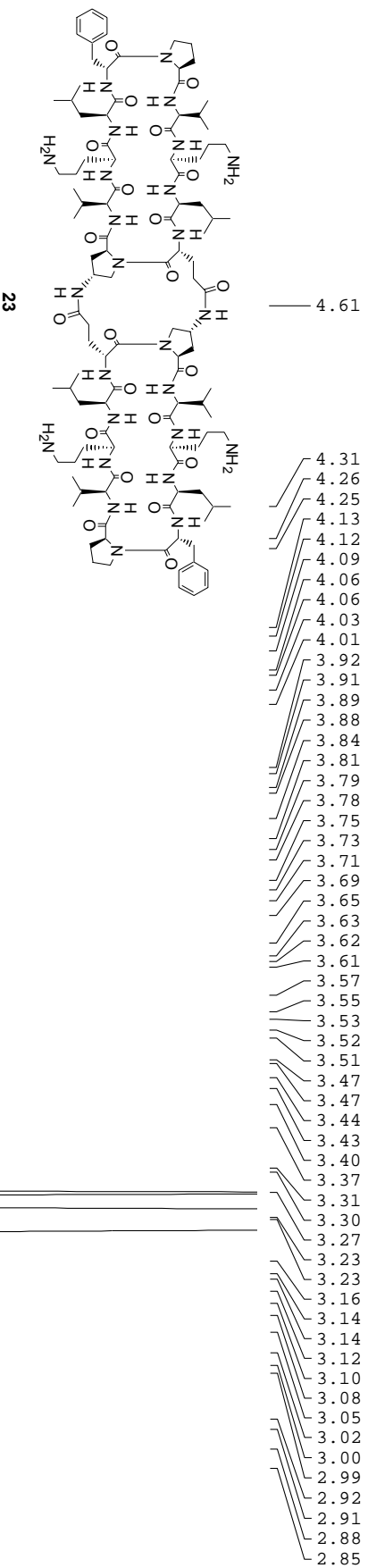
Current Data Parameters	
NAME	GJso72003
EXPNO	2
PROCNO	1
F2 - Acquisition Parameters	
Date_	20030723
Time	10:20
INSTRUM	avance400
PROBHD	5 mm BBI 1H-BB
PULPROG	zgpr
TD	19228
SOLVENT	MeOD
NS	128
DS	0
SWH	4807.692 Hz
FIDRES	0.250036 Hz
AQ	1.9997621 sec
RG	228.1
DW	104.000 usec
DE	8.50 usec
TE	300.0 K
D1	1.00000000 sec
d12	0.00002000 sec
d13	0.00000300 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 usec
PL1	4.00 dB
PR1	58.19 dB
SFO1	400.131865 MHz
F2 - Processing Parameters	
SI	131072
WDW	400.1300369 MHz
SSB	EM
LB	0.50 Hz
GB	0
PC	1.00

dimer over turn 1h nmr av-400 ATM-bbigs probe



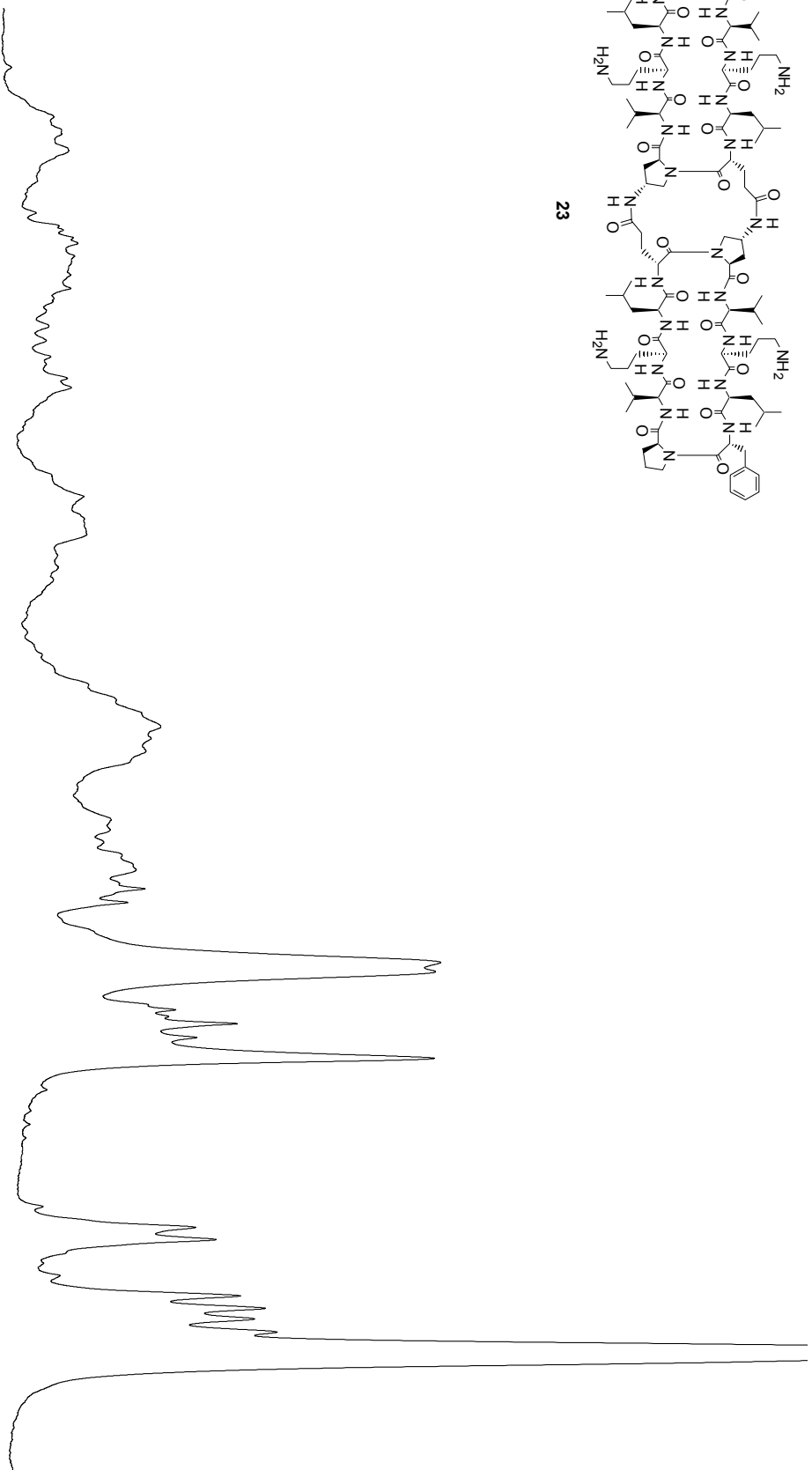
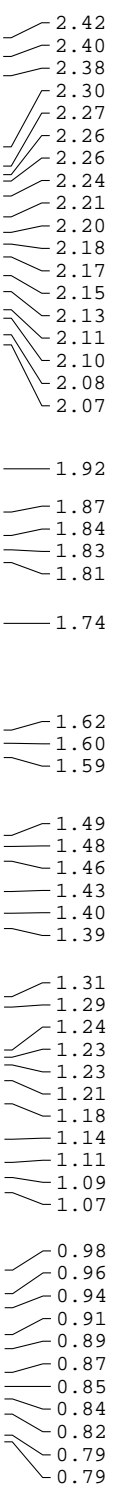
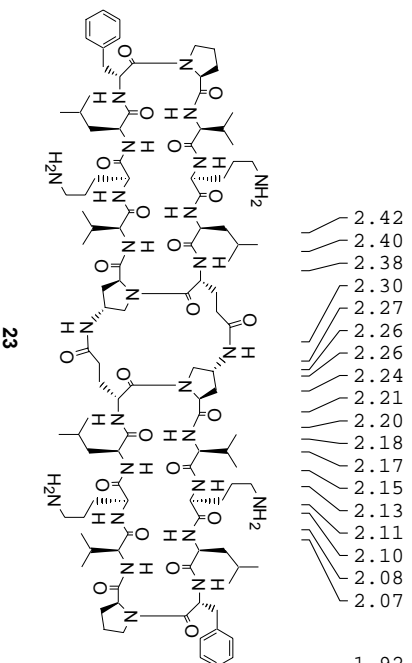
Current Data Parameters	
NAME	GJ5072003
EXPNO	1
PROCNO	2
F2 - Acquisition Parameters	
Date_	20030723
Time	10.20
INSTRUM	advance400
PROBHD	5 mm BBI 1H-BB
PULPROG	zgpr
TD	19228
SOLVENT	MeOD
NS	128
DS	0
SWH	4807.692 Hz
FIDRES	0.250036 Hz
AQ	1.9997621 sec
RG	228.1
DW	104.000 usec
DE	8.50 usec
TE	300.0 K
D1	1.00000000 sec
d12	0.00002000 sec
d13	0.00000300 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.20 usec
PL1	4.00 dB
PT1	58.19 dB
SFO1	400.1318663 MHz
F2 - Processing Parameters	
SI	131072
SF	400.1300389 MHz
WDW	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

dimer over turn 1h nmr av-400 ATM-bbigs probe



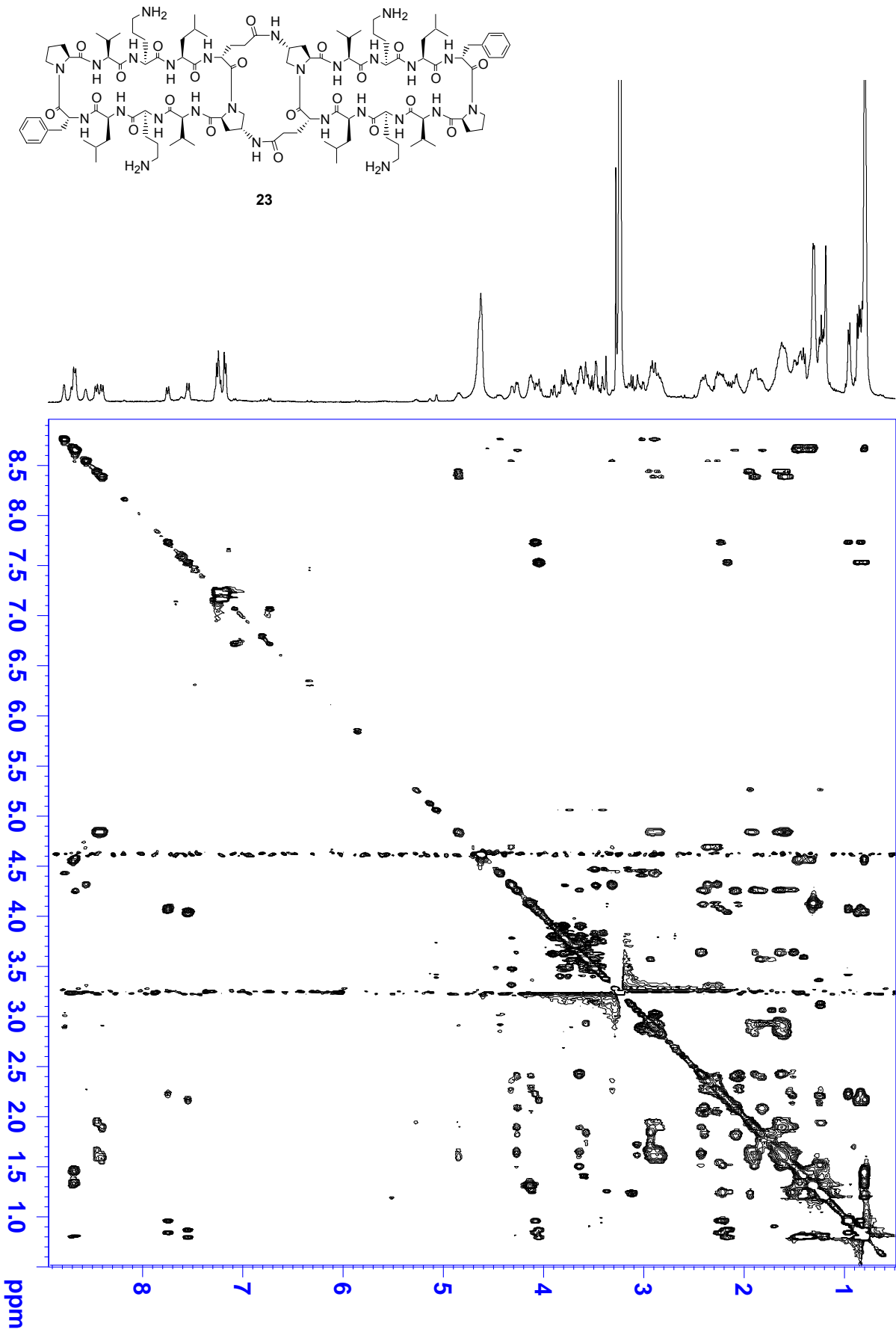
Current Data Parameters	
NAME	GJ9072003
EXNO	1
PRCNO	2
F2 - Acquisition Parameters	
Date--	20030723
Time--	10.20
INTERUM	avance400
PROBHD	5 mm BBI 1H-BB
PULPROG	zgpr
TD	19228
SOLVENT	MeOD
NS	128
DS	0
SWH	4807.692 Hz
FIDRES	0.250036 Hz
AQ	1.9997621 sec
RG	228.1
DW	104.000 usec
DE	8.50 usec
TE	300.0 K
D1	1.00000000 sec
d12	0.00002000 sec
d13	0.00000300 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 usec
PL1	4.00 dB
PT1	58.10 dB
SFO1	400.1318863 MHz
F2 - Processing Parameters	
SI	131072
WDW	400.1300389 MHz
SSB	EM
LB	0
GB	0.50 Hz
PC	0
	1.00

dimer over turn 1h nmr av-400 ATM-bbigs probe



Current Data Parameters	
NAME	GJ5072003
EXNO	1
PROCN0	2
F2 - Acquisition Parameters	
Date--	20030723
Time--	10.20
INSTRUM	advance400
PROBHD	5 mm BBI II-HB
PULPROG	zgpr
TD	19228
SOLVENT	MeOD
NS	128
DS	0
SWH	4807.692 Hz
FIDRES	0.250036 Hz
AQ	1.9997621 sec
RG	228.1
DW	104.000 usec
DE	8.50 usec
TE	300.0 K
D1	1.00000000 sec
d12	0.00020000 sec
d13	0.0000300 sec
===== CHANNEL f1 =====	
NUC1	1H
P1	7.30 usec
P11	4.00 dB
P19	58.19 dB
SFO1	400.1318863 MHz
F2 - Processing parameters	
SI	131072
WDW	400.1300389 MHz
SF	EM
SSB	0
LB	0.50 Hz
GB	0
PC	1.00

dimer over turn 1h nmr av-400 ATM-bbigs probe, cleanTOCSY Tmix=70msec



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Current Data Parameters
NAME G13s072003
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030723
Time 10.54

INSTRUM advance400
PROBHD 5 mm BBI 1H-2B
PULPROG cmlleapzg
TD 65536
SOLVENT CDCl3

NS 8
DS 256
SWH 4807.692 Hz
FIDRES 2.347506 Hz
AQ 0.2130420 sec
RG 2580.3
DW 104.000 usec
DE 8.50 usec
TE 300.0 K

TD 1.0000000 sec
D1 0.0300000 sec
d11 0.0000200 sec
d12 0.0000200 sec
d13 0.0000400 sec
D20 0.0006500 sec
INO 0.0002080 sec
I1 18
MCREST 0.0000000 sec
MCWRK 0.0099999 sec
STCNT 256

===== CHANNEL f1 =====
NUC1 1H

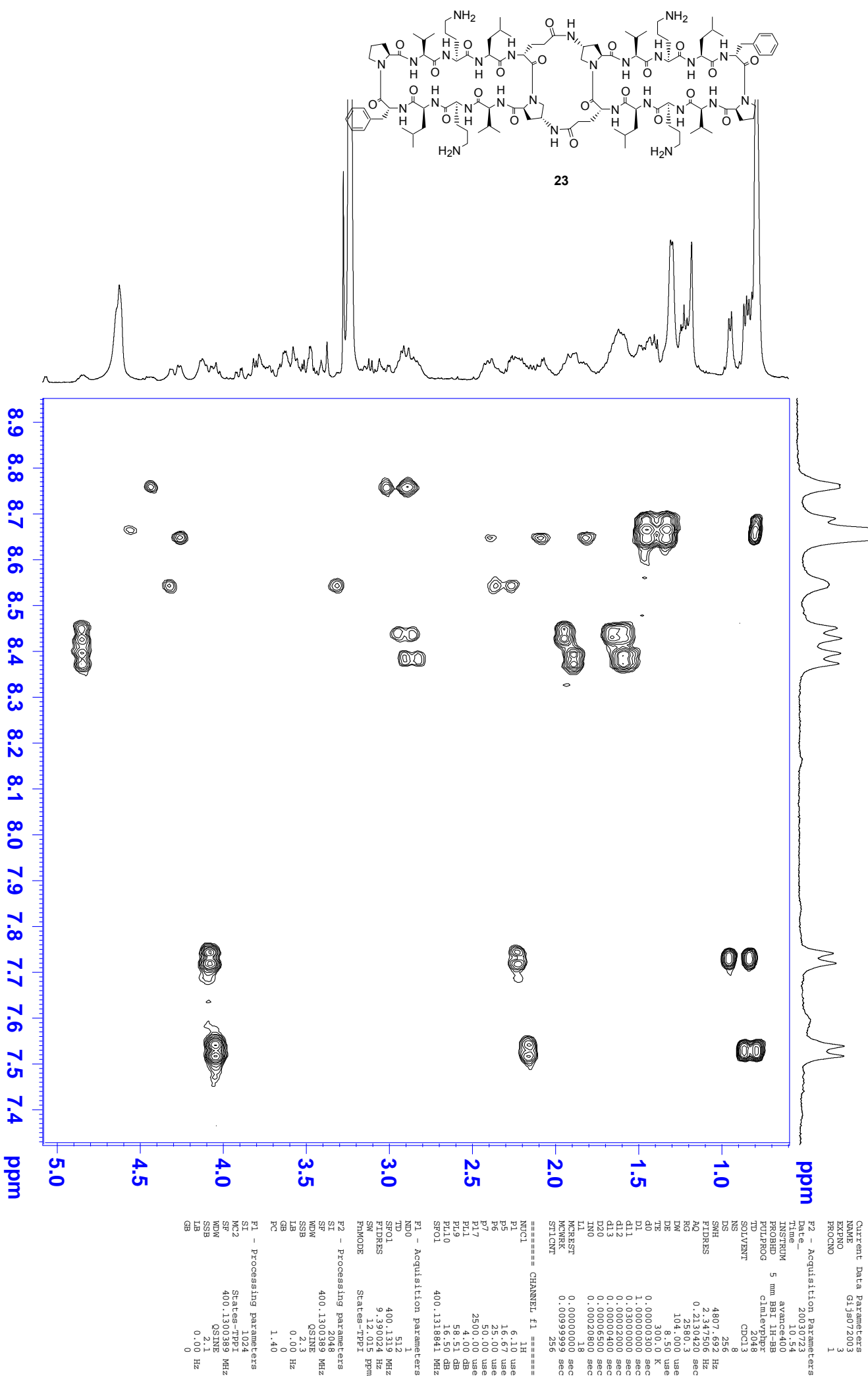
P6 6.10 usec
P5 16.67 usec
P7 25.00 usec
P17 50.00 usec
P11 2500.00 usec
P19 4.00 dB
P10 58.51 dB
SFO1 400.1318941 MHz

F1 - Acquisition Parameters
NUC2 1H
SFO2 400.1318 MHz
FIDRES 9.390024 Hz
SW 12.015 ppm
FMODE States-TPPI

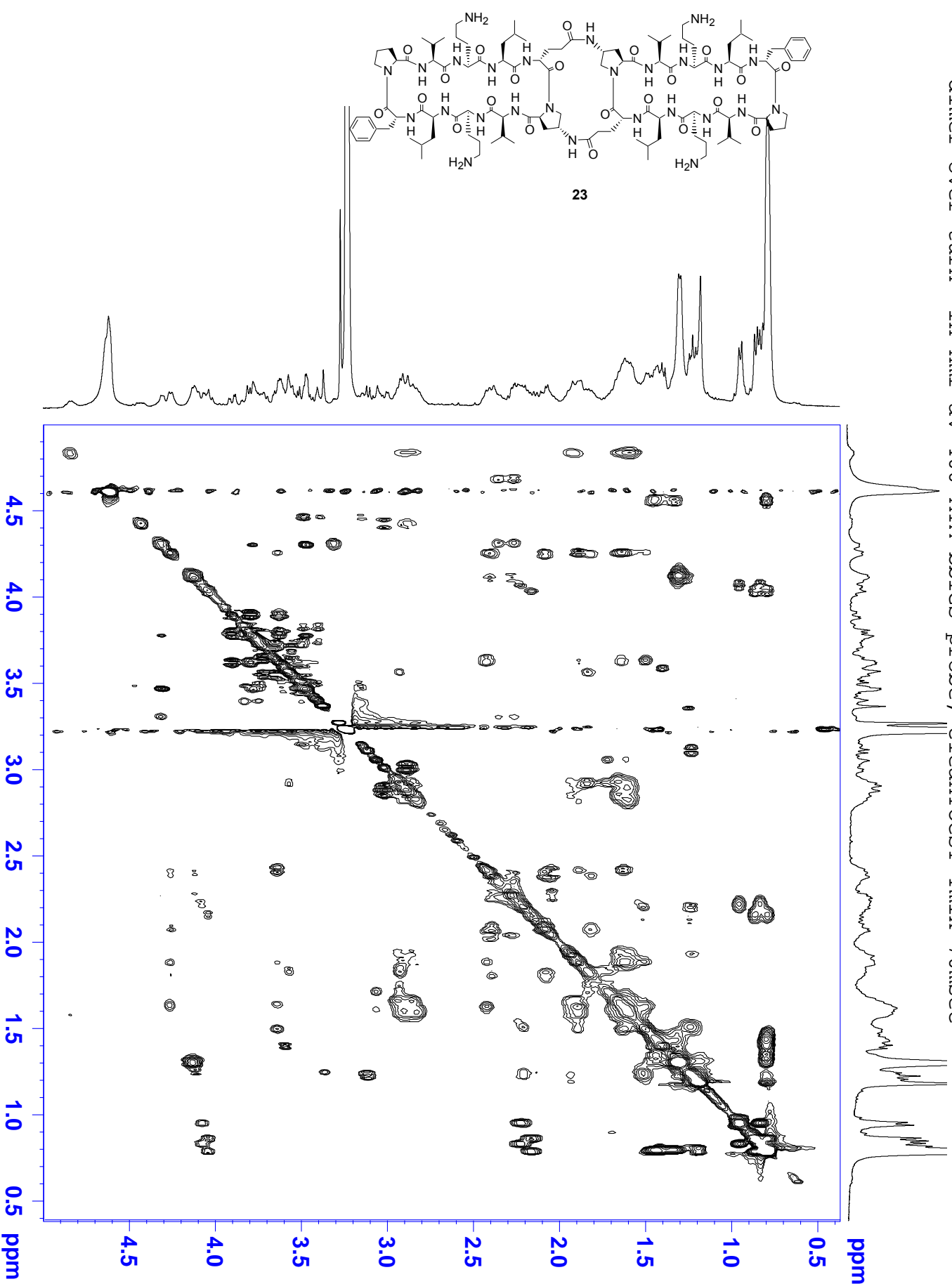
F2 - Processing Parameters
SI 2048
SF 400.1300389 MHz
WDW QSI
SSB 2.3
LB 0.00 Hz
PC 1.40

F1 - Processing Parameters
SI 1024
States-TPPI
MC2 400.1300389 MHz
WDW QSI
SSB 2.1
LB 0.00 Hz
GB 0

dimer over turn 1h nmr av-400 ATM-bbigs probe, cleanTOCSY Tmix=70msec



dimer over turn 1h nmr av-400 ATM-bbigs probe, cleanTOCSY Tmix=70msec



Current Data Parameters
NAME G1js072003
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030723
Time 10.54
INSTRUM avance400
PROBHD 5 mm BBI 1H-2B
PULPROG clineppzg
TD 65536
SOLVENT CDCl3
NS 8
DS 256
SWH 4807.692 Hz
FIDRES 2.347506 Hz
AQ 0.2130420 sec
RG 2580.3
DW 104.000 usec
DE 300.0 K
TE 300.0 K
D1 0.0000000 sec
d11 0.0300000 sec
d12 0.0000200 sec
d13 0.0000400 sec
D20 0.0006500 sec
INO 0.0002080 sec
I1 18
MCREST 0.0000000 sec
MCWRK 0.0099999 sec
STLCNT 256

===== CHANNEL f1 =====

NUC1 1H
P1 6.10 usec
P5 16.67 usec
P6 25.00 usec
P7 50.00 usec
P17 2500.00 usec
PL1 4.00 dB
PL9 58.51 dB
PL10 16.50 dB
SFO1 400.1318941 MHz

F1 - Acquisition Parameters

NUC2 513
SFO2 400.1319 MHz
FIDRES 9.390024 Hz
SW 12.015 ppm
FMODE States-TPPI

F2 - Processing parameters

SI 2048
SF 400.1300389 MHz
WDW QSI
SSB 2.3
LB 0.00 Hz
GB 0.00 Hz
PC 1.40

F1 - Processing parameters

SI 1024
States-TPPI
MC2 400.1300389 MHz
SF QSI
WDW QSI
SSB 2.1
LB 0.00 Hz
GB 0